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Glimpses into

The Past of the Dyeing Industry

By CARL C. CURTISS

TWENTY-FIVE hundred years before the birth of Christ an Egyptian took out a roll of papyrus, still preserved, and wrote: "The dyer has his fingers reeking, and their smell is that of fish spawn. His two eyes are oppressed with fatigue, his hand works incessantly, and as he spends his hours cutting out rags, he curses the garments."

In the second century A. D., in his famous "Lives," Plutarch makes this remark: "We often value a work and despise its creator, as for example in the case of salves and purples; we derive pleasure from them, but we regard the dyers and makers of salves as vulgar and narrow minded fellows."

Some thirteen centuries after Plutarch wrote, or in the period around the year 1500, it was necessary in Germany to serve five years as an apprentice before one could become a master dyer. And the hours of an apprentice dyer were from four in the morning to seven at night.

Four thousand years of the dyeing craft are spanned by these three records and in each year and in every country the dyer's life was hard. One exception should be mentioned, however. In Persia the dyer was held in great respect and was a pampered citizen. An old Persian legend relates that Christ was a dyer and He was the patron saint of the Persian dyer during the Middle Ages.

The shine on a man's shoes and the shine on a man's suit are two vastly different things in the scale of human values. So with the smell of the purple fish, the cause of the stigma fixed on the dyer's craft in ancient times. This mollusk which secreted the drops of precious fluid used to make the Tyrian purple left an odor on the person of the dyer which damned him in the eyes of his fellows, but the same odor in a dyed garment was the mark of supreme luxury, of eminent social distinction and usually the index of royalty.

In ancient China and among the Jews of Biblical times there seem to have been no professional dyers. In these countries dyeing was a home industry and the women dyed in the skein before their deft fingers spun the fabrics, each household being supplied its finished garments within the walls of the home. The Chinese did not wear wool except as a substitute for fur; thus cotton and silk were the only materials which they dyed. Silk is invariably referred to by writers of other ancient countries as "wool combed from trees," and a pound of silk in imperial Rome was worth its weight in gold.

But one of these nations of antiquity had neither a home industry in dyeing nor professional dyers. The Spartans with traditional austerity expelled from their country those who made perfumes as being wasters of oil; and those who dyed wool, as being "destroyers of the whiteness of wool."

It should be remembered that among the ancient Greeks and Romans dyeing was a luxury trade. The great mass of people wore undyed clothing and colors brightened the garments of only the upper classes. This widespread use of undyed material gave rise to an immense industry in scouring the easily soiled material. One can readily perceive the profound sociological consequences of such a practice; the sight of a dyed garment must have filled the lowly with a painful consciousness of their own humility.

Those primitive ancestors of the modern European and American, the barbarian tribes to the north who were to topple the decadent empire of Rome, had a love of color no less intense than the legions who so long held them at bay. The ancient German tribes, oddly enough, did not share the love of bright colors which is so typical of the savage. Black was their favorite color and was worn on gala occasions while brown was the dress of their common people. And the neighboring Gauls who roamed the wilderness approximating the present boundaries of France and Belgium showed a perverse streak by making purple, the color reserved for the great ones

of the then civilized nations, the mark of their inferior people. This Gallic purple is supposed to have been dyed from the ripe privet.

The ancient Briton was exceedingly vain and his favorite color was red. This hue appeared prominently in his attire which usually consisted of striped cloth with little squares of many bright colors. In his more primitive state, ages before his written history begins with the invasion of Caesar, the Briton had painted his face and body like our own Indians. The Romans found him dressed in sleeved jacket, trousers, hat and cloak, the cloth being a coarse felt made of wool pressed firmly and of such thickness and strength as to be considerable protection against the thrust of a sword. The colors for these garments were obtained from dyes extracted from barks of various trees and from lichen.

For five centuries after the fall of Rome it was the lot of the Byzantine Empire to preserve the remnants of that stricken civilization and it was only in Byzantium that the art of dyeing continued in its former splendor. The beautiful fabrics made in this empire of the east found an eager market in every princely and ecclesiastical establishment and in every abode of wealth in Europe. There the dye plants madder and woad were cultivated and there the purple fish continued to yield its precious color.

But the supreme achievement of the Byzantine Empire of importance to the dyeing industry was the introduction of the silkworm in Europe, an accomplishment deserving to rank as one of the greatest of all peacetime victories of one nation over another. For the Chinese were jealous of the precious insects which fed on the leaves of their white mulberry trees and no people had succeeded in transporting them alive from the Celestial domains. But during the sixth century two missionaries at Nanking, conspiring with the Emperor Justinian, hid a quantity of the eggs of the silkworm in the hollow of a cane and were successful in preserving them during the nine months' trek across the breadth of Asia, so that they were hatched and the worms and butterflies survived in sufficient numbers to propagate the forbears of the flourishing silkworm industry of southern Europe.

With the decadence of the Byzantine Empire dyeing fell largely into the hands of the Jews, not only in the East but in Italy as well. In Jerusalem they acquired a complete monopoly of the industry from the king and in the eleventh century frequent mention is made of revenue accruing to the pope from taxes levied on the Jews for the operation of their dye-houses. At this time princes sometimes maintained dyeing establishments and employed Jews for their workmen. It was the Jews who started the cultivation of indigo in Italy, a plant heretofore grown only in India, and which was later to replace completely the native woad.

The Venetians were the first people to wrest supremacy in the art of dyeing from the Jews. They continued the use of indigo and imported Brazil wood from India. The same excellence that characterized Venetian dyestuffs also marked their textiles and these marvellous products enjoyed a demand throughout Europe comparable to that previously held by the Byzantines.

During the thirteenth century the control of the dyeing industry passed from the hands of a single people and the art flourished throughout Europe, especially in Holland, France and Germany. English dyers were unable to compete with those of the continent.

But while the dyeing industry had passed from the control of a single people, the profits from one of its most lucrative branches were destined to be centered in one institution—the Church. It was Pope Pius II who in the fifteenth century gained control of the richest alum mines in Europe, the Tolfa mines, thereby acquiring for the Church ownership of the principal source of the mordant which was indispensable for the attainment of perfection in the application of contemporary dyestuffs. A later pope has told the story of this discovery and acquisition of the mines in detail in a book published in 1614.

An Italian dyer, John de Castro, had acquired a fortune by dyeing cloth made in Italy in his dye works at Constantinople where there was an abundant supply of alum. Castro studied the methods employed by the Turks in the manufacture of this alum and also paid close attention to the nature of the stone from which it was made. When Constantinople fell to the Turks in 1453, Castro lost his entire property but escaped with his life to Italy where he was indemnified for his losses by his relative Pius II, who raised him to the office of commissary-general over all revenues of the Church.

Whether Castro was fired with a zeal to enrich the Church or to avenge himself against the Turk for the loss of his dyeing business cannot be told, but he travelled the hills and mountains of Italy searching the earth for alum stones. At length he discovered some in the neighborhood of Tolfa, and having experimented with them and found them to contain excellent alum, he returned to the Pontiff with the news.

"I announce to you a victory over the Turk," said Castro, "he draws yearly from the Christians above three hundred thousand pieces of gold, paid to him for alum with which to dye wool of different colors, because none is found here, but a little at the island of Hiscala, near Puteoli, and in the cave of Vulcan at Lipari, which having been exhausted by the Romans, is now almost destitute of substance. I have, however, found seven hills, so abundant in it, that they would be almost sufficient to supply seven worlds. If you will send for workmen sufficient, and cause furnaces to be constructed, and the stones to be calcined, you may furnish alum to all Europe; and that gain which the Turk used to acquire by this article, being thrown into your hands, will be to him a double loss."

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Rayon Dyeing and Finishing—2

By **B. L. HATHORNE**
Consulting Textile Chemist

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CHAPTER I—METHODS OF RAYON PRODUCTION

(*Continued from page 172, March 13th issue*)

Nitrocellulose Process

The nitrocellulose or Chardonnet process of artificial silk manufacture depends upon the solubility of nitrocellulose in a mixture of alcohol and ether. Purified cellulose is nitrated with a mixture of nitric and sulfuric acids after which it is dissolved in a mixture of alcohol and ether to form collodion. The collodion so formed is forced through spinnerettes into a spinning chamber where the ether evaporates thus partially precipitating the collodion and forming a continuous filament. The filaments are drawn from the spinning chamber and are wound upon a bobbin that revolves in a water bath. The water extracts the alcohol and leaves precipitated nitrocellulose yarn upon the bobbin.

The precipitated yarn is twisted, skeined and treated in a sulfhydrate denitrating bath which effectually removes the nitrate radical from the yarn. The denitrated yarn is subsequently washed, bleached and treated in a finishing bath.

The chemical reactions of the nitrocellulose process appear simple, but are in reality highly complex. The sequence of the reactions that result in the removal of the nitrogen groups from nitrocellulose are so involved that no attempt will be made to explain them in this article. We will content ourselves with considering only the starting and the ultimate product, omitting the various intermediate reactions that must take place and that must be accurately controlled. The generally accepted formula covering the production of nitrocellulose yarn are:—

Nitration:—

$$C_6H_8O_3\begin{matrix}/OH\\ \backslash OH\end{matrix} + 2H\,NO_3 \rightarrow C_6H_8O_3\begin{matrix}/O\,NO_2\\ \backslash O\,NO_2\end{matrix} + 2\,H_2O$$

Denitration:—

$$C_6H_8O_3\begin{matrix}/-NO_2\\ \backslash -NO_2\end{matrix} + 2\,Na\,SH \rightarrow C_6H_8O_3\begin{matrix}/OH\\ \backslash OH\end{matrix} + 2\,Na\,NO_2 + 2\,S$$

$$2\,NaSH + 2S \rightarrow Na_2S_3 + H_2S$$

Nitrocellulose manufacture presents many interesting problems to the physical chemist; for example, minor changes in temperature, concentration, and time of nitration affect the rate of solution, total solubility and the viscosity of the spinning solution, which however must remain constant in order to insure the production of uniform yarn. Similarly, the physical properties of the finished yarn are greatly affected by conditions observed during denitration.

The physics of the spinning process is particularly interesting, because of the slow rate of coagulation of the nitrocellulose. When the collodion emerges from the spinnerette, it is homogeneous in structure and can be considered as a true plastic. As the ether evaporates during spinning, the yarn gradually assumes a definite structure. The rate of spinning, the rate of solvent evaporation (controlled by temperature and solvent used) and the stretch to which the yarn is subjected during spinning all have their effect upon the physical properties of the resultant yarn. The simultaneous control of these various factors is the most interesting and the most difficult part of the nitrocellulose process.

Purified cotton linters are treated with a mixture of sulfuric and nitric acid in huge nitrators. The actual nitrating is of course the function of the nitric acid. The sulfuric is necessary to absorb the water formed by the reaction of nitric acid with alpha cellulose.

At the completion of the nitrating process, the excess acid is removed and the entire charge is "drowned" in a water bath. The nitrated cotton is then continuously rinsed until all traces of acid have been removed, and extracted in centrifugal extractors, until it contains a definite moisture content.

The nitrated cellulose is slowly added to a mixture of alcohol and ether in huge mixing tanks where it is continuously churned and mixed for a long period of time. The thick transparent mass of collodion thus produced is forced through a series of filters into storage tanks, where it is held until needed for the spinning operation.

Collodion is pumped from storage tanks through a series of filters into small individual pumps that force the collodion through very minute glass capillaries into the spinning chamber. The newly formed filaments are

pulled through the spinning chamber over a guide and are wound upon bobbins, the surface speed of which is materially greater than the rate of flow of collodion from the spinnerette.

In the spinning chamber, the temperature, humidity and solvent content of the air must be kept absolutely constant in order that the rate of "setting" of the yarn shall be uniform. The gasses from the spinning chamber are continuously drawn from the chamber and run through a condenser where they are recovered for further use.

The temperature as well as the alcohol content of the water bath in which the filaments are wound must be kept absolutely uniform. This rinsing bath is continuously removed and carried to a recovery system where the alcohol is extracted and put back into process.

After the spinning operation, the nitrocellulose yarn is carried to the twisting room where it is continuously twisted and wound on spools.

The twisted nitrocellulose yarn, still in a wet condition, is carried to the skeining room where it is wound into yarn skeins for the denitrating process.

Nitrocellulose skeins, still in a wet condition, are carried to the denitrating department where they are immersed in a water bath containing sodium sulfhydrate—the denitrating medium. The skeins are treated in the sulfhydrate bath until the nitrogen content of the yarn is reduced to a mere trace. This process is much more involved than one might suppose, due to the number of intermediate reactions that take place during the denitrating treatment.

Following the denitrating bath, all traces of denitrating liquor are rinsed from the skeins, after which they are given a mild bleaching treatment, followed by a treatment with a minute quantity of finishing oil.

From the finishing bath, the skeins are conducted to the drying room where they are continuously dried and conditioned in huge specially constructed driers.

The dried skeins are automatically weighed to assure the consumer of uniform denier. They are then carried to the inspecting department where each individual skein is carefully examined by skilled inspectors, who grade the skeins according to their condition, number of broken ends, etc.

After grading, the skeins are packed and stored in warehouses, the humidity of which is carefully controlled, until they are shipped from the plant.

Production of Cellulose Acetate Rayon

The production of cellulose acetate rayon depends upon the solubility of cellulose acetate in acetone. Purified alpha cellulose is acetylated under carefully controlled conditions with acetic acid and acetic anhydride to which a catalyst has been added. The acetylated cellulose is "drowned" in water which causes the cellulose acetate to precipitate in the form of small cellulose acetate crumbs. The precipitated cellulose acetate is washed free from all traces of acid, dissolved in acetone, filtered, forced through metal spinnerettes into spinning chambers where the acetone is evaporated. The precipitated cellulose acetate rayon is continuously drawn from the spinning chamber and wound on bobbins.

The generally accepted formulae covering the production of cellulose acetate yarns are:—

(1) Acetylation:—

$$C_6H_7O_2(OH)_3 + 3\,(CH_3CO)_2O \rightarrow C_6H_7O_2(O{-}COCH_3)_3 + CH_3COOH$$

(2) Saponification:—

$$C_6H_7O_2(O{-}COCH_3)_3 + HOH \longrightarrow C_6H_7O_2(OH)(O{-}COCH_3)_2 + CH_3COOH$$

The production of cellulose acetate rayon is much simpler in many ways than is the production of yarn by the other processes. The only part of the process that is particularly interesting from the viewpoint of physical chemistry, is the actual spinning of the yarn.

The machines used for spinning cellulose acetate rayon are somewhat similar to the machines used for nitrocellulose with this exception:—the yarn does not come in contact with a bath of any sort as does nitrocellulose yarn. As the problems encountered in the spinning of cellulose acetate are somewhat similar to those of nitrocellulose, we need not discuss the spinning of cellulose acetate in detail.

After spinning, cellulose acetate yarn is twisted in the same manner as are other rayons. It is necessary that the relative humidity of the twisting room be maintained at least 90 degrees in order that static charges will not accumulate upon the yarn and machines during twisting.

In order to facilitate the twisting operation, as well as subsequent manufacturing operations, both in the rayon plant and in the plant of the consuming mills, a small amount of pure olive oil is applied to the yarn. The oil materially adds to the running properties of the yarn and assists in reducing loss from broken filaments, etc., to a low level.

After the twisting operation, cellulose acetate yarn is wound into skeins, upon cones, or on spools, according to the "put-up" desired by the ultimate consumer. This final operation as well as the twisting operation must be carried out in rooms having a very high relative humidity.

After being put into commercially acceptable form, the yarn is packed and stored pending shipment.

One outstanding advantage of the cellulose acetate process is the fact that the entire production of yarn leaving the mill is first quality. Tangled skeins, yarn containing broken filaments, etc., can be easily and quickly redissolved and respun into "A" grade yarn.

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Washington News Letter

(Special Correspondence from the Capital)

By **WALDON FAWCETT**

Textile Chemistry's Contacts

TEXTILE chemistry, and all the practical interests of the dyestuffs industry, have a stake in the first session of the 73rd Congress the proportions of which are not fully comprehended. On the one hand, and obvious in its extent, is the participation in the legislative responsibilities and problems of the hour. That is to say, the industry's share in the issue of unemployment relief, in the revision of the nation's banking system, in the fate of the gold standard, in the balancing of the budget, in the maintenance of the Open Door in the East. In short, in all the major elements of national policy that are vital to the restoration of normal trade and the return of prosperity.

This is the second of a series of articles written especially for the REPORTER on current events in Washington. The first appeared in our issue of January 16th. Others will be published from time to time as developments of interest to our readers take place.

Parallel to these liabilities of the extra session of the new Congress, stand a less conspicuous category the contact of which with the industry is even more intimate and with yet more potent influence. Here, for example, looms the question of what readjustments must be faced in Federal research work in behalf of textile chemistry at the National Bureau of Standards and other laboratories if there is a sweeping reorganization of the executive machinery of the Government. And if there is a horizontal reduction of 25 per cent in the cost of operating the Government. Wheels within the wheels of general legislation revolve toward the dyestuffs industry. If a general revision of the tariff at the special session is not a probability, piece-meal revision is always a possibility. If a General Manufacturers Sales Tax is a contemplation that is in disfavor with the new Administration, there remain the potential alternatives, as, for example, the proposed tax on interstate shipments and the visioned expansion of the special excise tax schedule to encompass, as "less essentials" or luxuries all the higher-price grades of wearing apparel.

Withdrawal of Government Competition

Along with the perennial propositions (such as Resale Price Fixing for Trade Marked Articles) which the 73rd Congress has inherited from the 72nd Congress, comes one fresh legislative nomination. This is the program to restrict or eliminate Government competition with private business. The Report of the Special Committee of Congress, which brought this matter to a head, was submitted too late for corrective action in the old Congress. Hence there wait upon the new Congress the rather drastic recommendations that the Government withdraw from competition with private enterprise and procure the commodities and facilities of all kinds which it requires for its use from the commercial and industrial world.

Involving upward of fifty different lines of industry, it is logical that this movement should have varied contacts, direct and indirect, with the color industries and continguous sections of the textile lines. One of the primary recommendations made by the Shannon Committee urges that the dyeing establishments maintained by the Army, Navy and other Departments, be restricted absolutely to actual service requirements, and under no circumstances be permitted to interfere with private business. A number of representatives of the dyeing and cleaning industry appeared before the Committee to protest the operations of the Government at the National Homes for Veterans, in the Panama Canal Zone, and at various forts, barracks, navy yards, etc. The accusation was made that the Government dyeing plants ignore major cost factors that must be reckoned with by private concerns in all competitive bidding.

Of interest to textile men is the recommendation that the operation of uniform and clothing plants maintained by the War Department at Philadelphia and the Navy Department at Brooklyn, be discontinued and the properties liquidated. Seventeen witnesses from the field of industry appeared before the Shannon Committee to protest manufacture by the Government not only of uniforms but of shirts, overalls, flags and other commodities in the textile classification. In consequence of the Congressional investigation, the manufacture of officers' uniforms has already been discontinued. Miscellaneous recommendations for the restriction of manufacturing by the Government call for discontinuance of the manufacture of ink and would put an end to the manufacture of paints at the Norfolk, San Francisco, Philadelphia, Charleston and other Navy Yards.

Manufacturing Chemists Protest

One of the significant phases of the movement to stay the industrial and professional hand of Uncle Sam concerns itself with research and laboratory tests. The Manufacturing Chemists Association of the United States formally protested to the Congressional Committee against the overextension of industrial research by the Government, to taking out of patents by Government employees, etc. With the issue thus joined, it is possible that in due course there will be formulation of a definite fixed policy to cover Governmental participation in technical research, experiment and chemical product development.

The chemists' representatives set forth that the present over-development of industrial research in Government laboratories is because of its "hidden character," apparently harmless but is actually, at times, "highly injurious" to industrial enterprises. It was conceded that much of this research by the Government was initiated by industries, associations or individual companies but the point was made that it has grown beyond the original intent or desired objectives.

Outstanding Evils of Industrial Research

Petitioners for curtailment of the scientific and technical explorative work of the Government have enumerated six outstanding evils which result from overexpansion of industrial research. First, was listed, the development from research into actual manufacture of products sold by the Government. Second,—The carrying out of research by Government agencies which interferes with the research of industrial companies and thus prevents the development of new enterprises under proper commercial auspices. Indictment Number 3, cites the retarding of industrial research by industrial groups cooperatively because of the activity within the Governmental bureaus.

Turning to the patent angle, the fourth criticism challenges the granting of patents as a result of Government investigation which are often taken out with the object of dedicating them to the public but which prevent exclusive development by industry on projects where large additional development cost is necessary thus actually retarding industrial development. The fifth count in the Chemist's bill of particulars declares that invention by Government workers frequently results in the owning of patents by individual Government workers who seek to exploit their inventions for their own private gain. The result of this is declared to be that the public not only pays for the research, but also pays a royalty to the Government investigators.

Finally, as the sixth flaw, in the existing system, the Association submits that the technical specialists of the Government become widely acquainted with industrial and trade practice and as a result are frequently consulted by industry. They then become direct competitors of professional consultants and deprive them of a legitimate part of their professional business. As a result, too, of such consultations, Government investigators often reveal trade secrets to the competitors of the company from which informaion has been obtained. This improper revealing of trade secrets is, with few exceptions, not done intentionally but nevertheless has serious consequences. In all this criticism of the existing policy of the Government, the critics recognize that a certain amount of technical research work is necessary in order to afford a basis for the Government's purchasing policies, etc.

Platform Proposed for Technical Participation

If the organized chemists can make their case, they hope to persuade Congress to lay down a few general principles,—duly defined by law, for industrial research. And to enforce these principles by the limitation of appropriations of funds for investigative and testing work if and when it is not feasible to dictate by law the precise limits to which Federal research may go. To this end it is recommended that all appropriations for industrial research be analyzed and reviewed to determine whether they involve a strictly governmental function, such as the national defense. In such cases curtailment is not suggested.

Allowing the exceptions indicated, all appropriations for industrial research merely as an aid to indutsry, are not considered to have sufficient justification to warrant continuation of such appropriations. Under the plan advocated, heads of departments or independent establishments would be required, in preparing requests for appropriations, to demonstrate the necessity for the continuance of such appropriations by proving that the investigations relate to necessary governmental functions; that they are fundamental in nature, not competitive with industry; and that no commercial outside institutional agency is available to conduct them if the Government does not do so.

Another recommendation from the same chemistry group is that extension at Washington to industrial research to the point of actual manufacture of goods, equipment, or other products for sale, should be prohibited and that such manufacture, even for the uses of the Government, should require specific authorization either of the President or of the Congress. By the same process of policy revision, the patent laws affecting the ownership of inventions would be corrected and investigations likely to lead to patentable inventions would be avoided. If a patentable invention did happen to result, the ownership of the patent would be so controlled as to stimulate development beneficial to the public and to prevent private exploitation.

Under the revolutionary ideals proposed to the Congressmen by the chemists delegation, free technical consulting services in any branch of science or technology should be limited to those nationally associated with the exercise of governmental function. Statistical, economic,

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PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

MARCH 27, 1933

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The Place of

Technical Men in the Textile Industry*

By H. H. WILLIS

Director, Textile Department, Clemson College, S. C.

THIS is a day and time in which mill conditions and practices never before questioned are being subjected to searching inquiry. Many changes both in technique and in policy are being brought about through the cooperative effort of practical and technically trained men. In many cases plants have been reorganized with a view to increased efficiency. Cost systems have been installed and each manufacturing process is being scrutinized with a view to decreasing cost without a sacrifice of efficiency or quality. The old slogan, "It has always been done that way," is being discarded in favor of the more progressive attitude expressed in the queries, "What is the most up-to-date method as applied to this process?" and "Is our method scientifically sound?"

In 1931, 62 per cent of the active spindles in the United States were in the cotton growing states, and these spindles consumed 79 per cent of the cotton used in this country. Thus the leadership of the textile industry has gradually swung to the South, and the South needs additional technically trained men to assist in making such leadership wise and efficient. Such leadership, to be sound, must be based upon scientific knowledge plus practical knowledge of manufacturing processes. The change in attitude of mill men toward technically trained men during the past ten years is noteworthy. Plants which were once unwilling to carry a college man on their payrolls, are now asking for them. In spite of the depression the demand for technically trained textile men from Clemson has increased rather than decreased. Practically 100 per cent of our last four classes of Clemson

*Presented at Meeting of Piedmont Section, Jan. 28, 1933.

textile graduates is in the textile industry. We thought that business conditions were poor in 1931. However, in June, 1931, the Clemson Textile Department placed 31 of its 32 seniors in less than four weeks after graduation and had some forty additional calls during the following six months. From the 1932 classes Clemson Textile Department placed 25 of its 28 graduates in the textile industry. I already have on file several inquiries for our graduates of next June.

How can technically trained men be of value to the industry? This can best be answered by a discussion of a few of the problems which have come to my attention during the past ten years during which time I have been connected with spinning tests and research work. Such problems are of like interest both to the manufacturer and to the textile chemist and dyer. The proper solution of these problems and the adoption of preventive measures for the future require the full cooperation of both practical and technically trained men engaged in the dyeing industry and in cotton manufacturing.

Size in Slashing

One problem is that of the size compound used in slashing—its preparation and technical control. Probably no two mills agree as to the time and temperature of cooking, and the kind and amount of size to be used on the warp for a given fabric. Poor sizing not only increases the cost of operation in weaving but also increases the amount of seconds. I have tested fabrics of similar construction and weight which differed from two to five per cent in the amount of size in the fabric. In weaving these fabrics, one had practically no shedding, while the other had an excess. The proper size mixture applied correctly means better running work and therefore a saving to the mill in production cost.

Regain

The factor of regain is given too little consideration. Many difficulties in manufacturing processes are directly attributable to inadequate control of regain. Regain and its control should be under the supervision of a technically trained man. [1]It requires constant study and supervision. Recently I tested some fabrics for one of the largest mills in the Carolinas. The regain in this fabric which was being shipped to the consumer was approximately six per cent though it could well have carried 7 to 8 per cent. Another angle of the problem of regain is that different varieties of cotton take up a different amount of regain under the same conditions. I have seen two varieties of cotton subjected to the same conditions for a given time, A showing 7 per cent regain and B showing 8 per cent. If these varieties have a different affinity for water, it is reasonable to assume that their dyeing qualities differ. A study of such problems requires the services of technically trained men. At least three of the most progressive mills in South Carolina have a Clemson textile graduate in charge of the test and research work for their respective plants. They have found these men a sound investment even during the depression. It is my belief that any mill of average size can well afford to have a technically trained man to look after the preparation and technical control of the size compounds used in slashing and to see that regain is properly controlled throughout the manufacturing plant. Improper sizing and inadequate control of regain are contributing their share toward putting manufacturing plants in the red.

Grade in Relation to Dyeing

As textile chemists you are especially interested in the dyeing properties of yarns and fabrics. What is the relation of grade and variety to these dyeing properties? The grade is determined mainly by three factors, color, foreign matter, and preparation. Of these three, color is most important to you. Although the factor of color in cotton may be somewhat negligible in some cases, it is very important in manufacturing fabrics or yarns for certain specific requirements. To cover the full range of color of cotton the U. S. Department of Agriculture has set up either working or descriptive forms for the various grades, which include Extra White, White, Blue Stained, Gray, Spotted, Yellow Tinged, Light Stained, Yellow Stained. Various problems in relation to cotton grading and varieties are discussed in detail during the four weeks' course in cotton grading given at Clemson College each summer beginning the second Tuesday in June.

In the so-called nine white grades, the low grades carry a small amount of blue or gray color which may be due to exposure in the field after opening. Tests show that neither blue nor gray cotton bleaches, nor takes on dyes in certain shades so satisfactorily as do the higher white grades. [2]These blue or gray cottons, when not mixed with other cottons, will produce a fabric which can be dyed uniformly, though the fabric may not have the same luster or brightness as one made from white grades. These blue or gray cottons if properly mixed or blended with white cottons will produce satisfactory shades for certain purposes. But the difficulty is in getting these different colors well mixed in manufacturing. If the mix is not thorough, streaked goods may result. Although such streaks may not show up in the gray or natural finish, they often appear in the finished product. I knew of a mill which was mixing some blue with white cotton to be woven into brassiere cloth. These two colors were not properly blended. This fact was not detected in the gray cloth as it left the mill but when it was dyed, streaks were very noticeable in the fabric because certain threads

[1]H. H. Willis, "The Importance of Regain and Its Control," July, 1932, *Cotton*.

[2]H. H. Willis, "Manufacturing Tests of Cotton of the White Grades of the Universal Standards for American Cotton," U. S. Department of Agriculture Bulletin 1488, April, 1927.

had an excess of blue cotton which did not take up dyestuff in the same manner as did the white threads in the same fabric. As a result the mill had a large shipment rejected. Sometimes neither the mill man nor dyer may know the cause of a defect and each may attempt to pass the responsibility to the other. I knew a dyer who had similar complaints and had assumed that he must be responsible for the defects until he came to the Clemson Textile Department and observed some tests on these different grades finished at the same time and in the same plant.

The yellow tinged and stained cotton, affected by soil stain or frost, will bleach and dye about as satisfactorily as the white cottons and can therefore be mixed with white cotton in manufacturing with less likelihood of streaked goods in finishing.

Variety in Relation to Dyeing

Tests show that different varieties of cotton vary widely in physical properties, such as fiber length, strength, uniformity, spirality and size of diameters. Why should the dyer be interested in these factors? Because these factors largely determine the uniformity of yarn that may be produced, and the best fabrics can not be produced from yarns lacking uniformity in strength and diameter. Yarn irregular in diameter may produce so-called cloudy fabrics, fabrics which have thick and thin places. With such material the dyer is unable to produce a finished fabric uniform in finish and appearance. The thick places in yarn have less twist than the thin places; hence a different amount of dyestuff may be taken up. The thin places with excessive twist reflect the light differently from the thick places having less twist. I have had a number of unsatisfactory, finished samples of fabric submitted to me, the defects of which were due to irregular yarns. When such fabrics are rejected, the manufacturer must take the loss. In many cases the manufacturer naturally assumes that the trouble was caused in the dyeing and finishing processes, whereas the difficulty may be traced directly to the variety of cotton used or to the mix used in the opening room. It is in locating such weaknesses in the manufacturing process that the technically trained man is of value.

I have had a number of examples of lack of uniformity in various fabrics but the most striking ones have been in window shade material. Such fabrics, as you know, are under the most rigid examination and small defects show up readily because the fabric is between the observer and the source of light.

The effect of this lack of uniformity in yarn diameter is well illustrated by a case which came up some months ago when I was called upon to assist in settling a controversy in connection with a fabric which contained thick and thin places. The manufacturer who wove the cloth attributed the defects in the fabric to imperfect yarn and assumed that either the spinning or finishing process was responsible. Accordingly, the case was first taken up with the yarn manufacturer; he attributed the trouble to the character of the cotton; the cotton buyer in his turn placed the responsibility upon the boll weevil poisoning used on the cotton plants. Upon analyzing the fabric, it was evident that it was a case of mixed yarn. Fillings from two different plants were being used in this particular fabric, one being much better in quality than the other. The filling in one case was very uniform, whereas in the other it was very irregular, being "thick and thin" as termed by the mill man. These bobbins of filling were thrown together and used as they came to hand. Thus the better filling yarn often alternated with that of inferior quality. The difference was, of course, very evident when seen side by side in the finished product. The technically trained man should foresee and prevent such occurrences.

Different grades and varieties of cotton lose different amounts of strength in the bleaching process. Loss in strength does not necessarily follow the grade of cotton. Two varieties of practically the same grade may vary widely in loss of strength when bleached. To illustrate: Lots A and B are the same grade and staple, and bleached at the same time under the same conditions. Lot A lost 4 per cent while Lot B lost 10 per cent. Apparently this difference is due to a difference in the variety of cotton used.

Bleaching does not necessarily reduce the strength of warp and filling in the same proportion. Mills frequently use a different variety or character of cotton in filling from that used in warp yarns. To illustrate: Sample C lost 5 pounds warpwise when bleached while the filling lost 12 pounds. The construction and the amount of twist may be responsible for part of this difference but part is attributable to the variety of cotton used.

Not all defects in dyeing and finishing are due to the manufacturing process and the grades and varieties used. Many defects are traceable to the dye house. Many of these defects can be avoided by the proper study and cooperation of the dyer and manufacturer.

May I tell you what Clemson Textile School is doing to help you solve your problems? You probably know that Clemson has by far the largest textile school in the South. Of the 175 students in the Clemson Textile Department, some 50 are specializing in textile chemistry and dyeing, which division is under the able leadership of Dr. Eugene R. Manning whose associate is Mr. A. R. Macormac. Realizing the effect of mixing and blending of cotton and the part that grade and variety of cotton play in dyeing and finishing, we have now incorporfated in our four-year textile chemistry and dyeing course the subjects of cotton mixing, cotton grading, and fabric design and construction. Thus these boys, when they come to your plants, will be better able

to assist you in tracing some of these defects in finishing. Our other 125 textile students are specializing in textile engineering, textile industrial education, and weaving and designing. We have also equipped a modern testing laboratory in which has been installed both humidifying and dehumidifying equipment. In this laboratory both our textile engineering students and our textile chemistry and dyeing students may study and test accurately the relation of different grades and varieties to the strength, dyeing and finishing properties of yarns and fabrics.

The demand for technically trained men for technical analysis and control in the textile industry is bound to increase with the return of prosperity for even then the margin of profits will not be sufficient to permit hit and miss methods and approximations.

WINTER MEETING, SOUTHEASTERN SECTION

THE winter meeting of the Southeastern section was held February 4th at the Colonial Hotel, LaGrange, Georgia. There were 80 members and guests in attendance.

During the business session there was a lively discussion of methods and means to secure corporate memberships through this section. There was real interest shown on this matter and every member was asked to talk the matter over with his mill officials and some action will be taken on it at the spring meeting to be held in Columbus, Georgia, April 29th.

Members and ex-members were urged to pay up their dues and become reinstated.

E. A. Fiemster, chairman, introduced Mr. Hatton Lovejoy who gave a splendid address of welcome.

The chairman then introduced W. F. Christman, Russell Manufacturing Company who spoke on "Dyeing and Processing of Knit Goods." The speech was highly instructive and Mr. Evans of the Newnan Hosiery Mills led the discussion.

The Dyeing and Processing of Knit Goods*

By **W. F. CHRISTMAN**
Russell Mfg. Co.

MR. Chairman and Gentlemen:

The subject assigned me is one that very few of the members are interested in, so I will only give a brief outline as related to cotton knit goods only.

The dyeing and processing of knit goods is an industry almost exclusively related to the manufacture of knit underwear. The knit cloth dress trade using almost exclusively dyed yarns knit in combination so that when the cloth comes from machines, it requires very little finishing before being cut into garments.

There are very few dyeing and finishing plants devoted exclusively to knit goods in the United States. You will find practically all of these located in Pennsylvania and principally in and around Reading. This was due to the fact that there were hundreds of small knit underwear mills located in middle and Eastern Pennsylvania, most every community having a mill. These mills knit the cloth, send it to the bleachery to be bleached, and when returned, cut and finished into garments. This saved these small mills the expense of maintaining a boiler plant, bleaching and dyeing equipment.

Today with few exceptions every underwear plant has its own dye house. There are very few knit underwear mills on cotton in Pennsylvania.

I will start with the yarn, so that you can get a clear understanding of the process: Knitting is a system of loops joined together to make a fabric.

First, the yarn is of very soft twist, the theory being to have just enough twist to hold the fibers together. The yarn is put up on cones for the knitting machines. The yarn has been thoroughly conditioned before knitting, no size or anything else being used.

Circular knitted cloth comes in tubular widths from 10" up to 36" in diameter and weighs from one yard up to six yards to the pound, depending on the width, fabric and size of yarn used. Cloth comes in rolls of one hundred to one hundred fifty yards, weighting twenty-five to seventy pounds to the roll.

In woven goods or yard goods as it is sometimes called, everything is reckoned by the yard, for instance, twenty to twenty-five yards to the dozen dress shirts. In underwear, everything is recorded by the pound as twelve pounds

*Presented at Feb. 4, 1933 Meeting of South Eastern Section.

to the dozen union suits; a dozen athletic shirts weighing two pounds, etc. So you see yardage doesn't play such a big part in knit underwear goods, as everything is reckoned by the pounds.

In the dyeing of knit goods, string washers or reel machines are used, which are machines similar to the rope soapers used to soap vat colors in piece goods, or slack washers, as they are some times called.

In boiling preparatory to bleaching there are three methods used; boiling in reel machines, in open kiers, or under pressure. The method used to boil depends on the outlay of equipment the mill has. Where the boil out is done in a reel machine, it is boiled for an hour to an hour and a half with one per cent caustic soda, one-half per cent Tri-Sodium phosphate and one-half per cent alkali soap, or penetrating oil. It is drained, rinsed and cooled, and the bleach liquor added to twadell two degrees. It is run forty-five minutes to secure a good white. The bleach liquor is then drained and the tub refilled, to which enough sulphuric acid is added to twadell three-fourths degree. This gives the goods what is known as a "Buck" sour which produces a very bright white finish. After this the goods are thoroughly rinsed and in one rinse is added a small amount, one-half per cent of Bi-Sulphite of soda to permanently free the goods of chlorine. Chlorine gas is the most satisfactory article to use in making up bleach liquor for knit goods.

Goods to be subsequently dyed are given what we call a half bleach, just bleached enough to obtain a good ground. A good uniform boil is absolutely necessary if you expect to get a uniform shade in dyeing.

Knit goods can be bleached with hydrogen peroxide or Sodium peroxide in kiers, but the cost makes this process almost prohibitive.

The most economical method of bleaching if you are fortunate enough to have equipment is to give the goods a pressure boil, wash down in the kier good and pull them out through a chemic squeezer, and pile in bins to bleach, after which it is put into reel machines and soured off, rinsed, and bluing added.

In the dyeing of knit goods the operation is carried on in a reel machine which holds from two to twelve rolls to the machine, weighing from one hundred to twelve hundred pounds per machine. The rolls are loaded and the ends tied or sewed together, each roll making an endless rope.

On heavy shades such as navys, browns, red, black, etc., the goods are boiled out for thirty minutes with Tri-Sodium phosphate, drained and washed, then the dyestuff is added when the goods and bath are cold. The steam is then turned on and the bath is gradually raised to a boil. It is allowed to boil for thirty minutes and salt is added. Continue the boil for twenty to thirty minutes to get good exhaustion of dye. The goods are then rinsed twice through cold water and unloaded.

On medium and light shades and also tints, where a good ground is required, the simplest method I know of is to give the goods a half bleach with Solozone in a reel machine which requires approximately an hour, then the goods are cooled down and dyestuff added, and run for fifty minutes before adding salt, then run ten minutes in the salt.

In using Solozone you can dye the goods first and then bleach, and I believe you get brighter shades by this method. I have noticed this to be true with certain dyes. Still another method used is the old Autogyp process where the dyeing and bleaching is done in the same operation, which is very satisfactory and is a time saver.

Just a few words about finishing of knit goods and I am through. After bleaching and dyeing the goods are extracted or run through squeezer rolls to remove all water possible.

There are two methods of drying employed, the loop dryer and the vertical tubes. The tube system consists of pipes four to eight inches in diameter, approximately sixteen feet long. Above these pipes is a rolling up attachment. The goods are pulled down on these pipes, and a spreader inserted between the top of pipe and rolling up rolls. The cloth is then pulled up and started rolling up. The hot air comes up through the pipe and drys the goods. Light weight cloth that does not require napping is ready to be cut into garments when taken from dryer. One medium and heavy weight fabrics that are napped, the process goes a step further. These goods are put over a turning machine and turned right side out after napping and folded. They are then ready to cut into garments.

As you will note there has been no mention made of softeners or finishing compounds in the finishing of knit goods. If you are careful in the preparatory process to use the correct amount of chemicals, you will find it will not be necessary to use any softeners to obtain a nice soft fabric.

I thank you.

Discussion

Question: What advantages are gained from use of vegetable oils or softeners on knitting yarns?

Answer: The only yarns that we use softeners on are the naphthols and then sulfonated olive oil is used.

Question: Do you ever knit solid colors from package dyed yarns?

Answer: We prefer raw stock dyed yarns for dyeing the greige goods in the piece.

R. H. Adams, Calloway Mills, gave a splendid talk on "Some Elementary Facts on Textile Testing." This was a very constructive talk and everyone enjoyed it.

C. B. Ordway, Alabama Polytechnic Institute, was asked to lead the discussion.

Proceedings of the American Association of Textile Chemists and Colorists

Some Elementary Facts in Textile Testing*

By **R. H. ADAMS**
Calloway Mills, La Grange, Ga.

HOW can testing affect the quality of the goods or the output? Such a question is a very natural and reasonable one. The vast number of raw materials, as well as the finished products, involved in many manufacturing operations necessitates the closest observation and the checking of each process regarding the tensile strength, stretch, elasticity, durability, weight, construction and the physical properties in general. There are good reasons for believing that any product can be made much better by the use of a well equipped and thoroughly maintained laboratory of physics and chemistry, as we are assured of the same quality day after day. Standard formulas for testing have been and are being developed so as to make and check specifications more intelligently, thereby removing one unnecessary source of friction between the consumer and the source of supply. It is with this aspect that I am concerned in this paper.

At the present time textile laboratories in the United States are divided generally into two groups—those operated as a part of a single mill organization and those operated for the common benefit of a group of mills, as a central testing laboratory. These units have grown to the point where they are of great importance to the success of the industry, since they are actually the only scientific agencies for originating new methods and securing economy in production, as well as improving the quality of products by experimentation. We have learned that all industrial operations present questions of a scientific nature since their special problems have led to the devising of special ways to solve them. We have learned that results of permanent value can rarely be obtained aside from extended experiments and practical research. Since the testing laboratory has been brought into close relation with the mill management and the mill departments industrial leaders have come more and more to see the value of testing their materials and thus saving time and money in the early determination of qualities and possibilities, whereas manufacturing methods had hitherto been based altogether on long years of practical experience and rule of thumb. Many failures resulted from lack of knowledge of the rigid requirements in the case of materials being produced against specifications.

In establishing a textile laboratory for routine and scientific testing three things are necessary. First, a skilled textile laboratory specialist who is acquainted with the scientific side of textiles as well as with mill machinery and its manipulations should be selected for the work. He must be tactful, as he will be brought into close relations with foremen and superintendents and must be able to get over to them his understanding of their problems and his sincere desire to assist them in solving these problems. Needless to say, he must be accurate, a close observer, a lover of his work, and so devoted to his equipment that every part of it is at all times in the best possible condition. As to his training, the excellent schools which we now have assure the ambitious young fellow a chance to begin on the technical side where his predecessor of only a few years ago might hope to be only after a life time of study and effort. My only comment here is that identical or similar equipment in school and industry will tremendously simplify the problem of adjustment; also that the elimination by the schools in early stages of students unsuited to laboratory work will earn the everlasting gratitude of industry and contribute greatly to its profit.

The laboratory equipped for testing of textile materials should be properly built, with plenty of room, insulated walls, abundant light, a complete set of machines for physical testing, and a complete set of analytical and chemical balances. I prefer an arrangement such as we have at Southwest LaGrange—a basement location, half-sunk under ground level. Machines, tables and equipment should be so arranged as to save time and energy. Especially important is it that all testing should be done in standard atmospheric conditions, since all results of value depend on the condition under which the tests are made. Naturally, the basic factors of relative humidity of cotton regain must be established under standard conditions before dependence can be placed on the results of the test. Cotton yarns and fabrics increase or decrease in tensile strength by six or seven percent for each one percent corresponding change in moisture. In this connection it is very important to observe the outside conditions at all times. Very often these conditions are rapidly swinging from one extreme to another. Variation in temperature, continued rainfall, extremely dry windy

*Presented at meeting, Southeastern Section, February 4th.

days, all these exert continuous influence toward fluctuations in weight and in tensile strength.

From my experience in testing of textile materials, I would have no faith in any breaking strength test or the weight in which the regain of sample being tested was not given. Regain in any material is determined by five variable factors: first, the nature of the material itself; second, the physical condition of the material, whether hard or soft twisted, whether in a multiple of plies or to the contrary; third, the relative humidity of the atmosphere to which it has been exposed; fourth, the temperature of the same atmosphere; fifth, the most important, the time of the exposure. It is said that Thomas A. Edison once remarked that a life time is too short to solve a problem containing more than three variables. Here we have five to start, and after that two others, both of which require skill in the operator after all preparations for a sample have been completed. You have the human and the mechanical elements in the preparation of the specimen of sample to be tested. It must be properly marked and lined, and the greatest of care must be exercised in adjusting it to the testing machine.

But your problems and variables do not stop here. After the finished product is baled, branded and placed in the storage room ready for shipment, you have at least one other problem. You have manufactured your goods under conditions which are desirable to produce quality, to preserve the natural moisture in the fiber being treated, enabling it as far as possible to maintain its original condition and thus preserving its strength and elasticity. But due to changing atmospheric conditions and the fact that the natural atmosphere has a continuous effect on textile materials, both during manufacture and afterwards, the possible variation in strength is so great that without careful provision for the influence of this factor a fabric might actually be rejected one day and, if there was a substantial change in the atmosphere overnight, be accepted the next day. All cotton yarns and fabrics are hygroscopic, and when a mass of hygroscopic material is exposed to an atmosphere containing water vapor, the process of absorption continues until the vapor pressure within the material and in the atmosphere have become equal. When this process ceases, a condition of hygroscopic equilibrium has been reached, whether it be in storage or in freight cars in transit in Maine, California or Georgia. A regain which would invite no fluctuation in weight after delivery, and which is practically attainable, would, of course, be satisfactory both to manufacturer and customer. It is doubtful, however, if any arbitrarily chosen standard would be acceptable throughout the American cotton industry until a proper foundation has been laid by close study and active cooperation between the buyer, seller, Sub-Committees of the American Society for Testing Materials, and other competent research societies.

For this reason all materials of current production being produced against specifications should be tested daily. They should be carried to the laboratory and thoroughly conditioned in an atmosphere which is as near a standard as is possible to maintain. After conditioning, the tests should be made under the same conditions as before and records of results should be made and compared with previous records. If this method is followed, a standard for all yarns and fabrics can be made and a semi-annual and a yearly average can be compiled to form a basis for a standard and to set a limit for reasonable tolerance. Any deviation from this can be promptly corrected at any time by checking closely with the mill, as the tensile strength should be kept at all times well above the margin required by the specifications.

In developing the routine work of a textile testing laboratory, there are two other important questions to consider in addition to the physical testing, namely, the analytical and chemical. The analytical process should be organized in such a way that the person in charge of a laboratory may take any sample received for duplication and analyze the weave and construction as to count, yarn numbers, weight, thickness, percentage of warp and filling, twist and determine the raw material that it will take to produce a like fabric. The chemical section should be equipped to handle any kind of finished materials that may be presented for extraction of rubber coated substances and enamellings, the dissection or identification of textile fibers and their quantitative determination in mixed goods, the determination of percentages in sizing compounds, and the judgment as to finishing materials used.

In conclusion, I should like to present a blue print of a laboratory and a blue print showing some of the various methods of testing fabrics and ducks. There are many methods in use in our laboratory, the blue print indicating fifteen of the tests most commonly used. This is to illustrate the importance of training a personnel. It requires considerable time to teach a person to read specifications, cut specimens as outlined, and make the fine adjustments mechanically necessary in changing from one set of specifications to another. Certainly in our day, when the tremendous speed of style and merchandise movements necessitates co-ordinated action in every industry in the country, it is vitally important that the textile mills have at their command every available scientific development and device for solving manufacturing problems promptly, accurately and economically.

I quote Pasteur: "Take interest, I implore you, in those sacred dwellings, laboratories. Demand that they be multiplied, that they be adorned. These are the temples of the future, temples of well being and of happiness. There it is that humanity grows greater, stronger, better."

Discussion

Question: Does the ASTM, D-13 Committee make recommendations for conditioning dyed or bleached materials?

Answer: Shorter period on lighter materials than heavy and about twelve hours on bobbins or dyed packages.

Question: What are your recommendations on type of sample rack?

Answer: Use movable type by all means, with r.p.m. of 2-10. This allows all sides to be properly exposed.

Question: Why was the Calloway laboratory placed in the basement?

Answer: We used the thermos bottle principle, cool air under building in summer time. The following illustration was given: On one hot August day, it was 97°F. out of doors and 81° in the laboratory.

Question: What is the time for sized yarns and three pound packages?

Answer: Sized yarns require 4-6 hours and the three pound package twelve hours at the minimum.

A rising vote of thanks was given Mr. M. T. Johnson and the program Committee.

Respectfully submitted,

CHARLES B. ORDWAY, *Secretary*.

ANNUAL MEETING, SOUTH CENTRAL SECTION

THE annual meeting of the South Central Section of the American Association of Textile Chemists and Colorists was held at the Read House in Chattanooga on the evening of February 18, 1933. Sixty-two members and guests were present.

During the dinner several entertainment features were presented by W. J. Kelly, Jr. These were enjoyed by all.

Mr. Noel D. White, Chairman, presided at the meeting. The minutes of the previous meeting were read and approved.

The following papers were presented:

"The Application of Sulfonated Oils," by A. J. Kelly, Burkart-Schier Chemical Co.

"Developed Colors," by E. P. Davidson, du Pont Co.

The annual election of officers was held, resulting in the unanimous election of the following:

Harold Schroeder, Dixie Mercerizing Company, Chairman.

L. E. Millikan, Aycock Hosiery Mills, Vice-Chairman.

Homer Whelchel, Central Franklin Process Company, Treasurer.

Andrew J. Kelly, Burkart-Schier Chemical Company, Secretary.

G. H. Dubois of the W. B. Davis & Son Company was elected a member of the Council of the Association.

Mr. Noel D. White was given a rising vote of thanks for his very successful term of office.

The report of the treasurer was read, showing that the Section had made a slight gain in finances during the year.

Respectfully submitted,

A. J. KELLY,
Secretary.

The Application of Sulfonated Oils*

By **ANDREW J. KELLY**
Burkart-Schier Chemical Co., Chattanooga, Tenn.

A CONSIDERABLE number of inventions and discoveries of interest and importance to the textile industry have been developed within a comparatively short time. These new developments speak for themselves—there is no need to mention them here. We are all interested and excited by what is new and novel; this is quite natural and more or less necessary if we are to keep abreast of the times. But in greeting the newcomer there is sometimes a tendency to overlook that which is already with us, meritorious though it may be. This latter statement might perhaps be applied to many things with which we are familiar—I want to apply it here to the sulfonated oils, and in particular to sulfonated castor oil, or Turkey Red Oil.

"One of the earliest organic molecules of immense importance to the dyer was Turkey Red Oil, discovered in 1875." That statement is taken from a paper presented by R. E. Rose of the DuPont Company at the recent national meeting in Greensboro. Government statistics state that in 1929 nearly twenty million pounds of Turkey Red Oil were consumed in this country, but this figure represents only a fraction of the true consumption.

*Presented at annual meeting, the South Central Section, Feb. 18, 1933.

However, when we consider the truly amazing properties of the modern sulfonated oils the wonder is that the quantities are not much greater, as it seems assured they will be.

With sulfonated castor oil, and to a lesser extent with other sulfonated oils, practically every type of water-insoluble solvent or lubricant can be solubilized or emulsified. This fact is of great importance in the wet finishing of all varieties of textiles, for it makes available a large number of valuable assistants for the conditioning of yarn for weaving and knitting and for the processes of scouring, dyeing, bleaching and finishing. Sulfonated oils give a finish on textiles that is difficult to duplicate. In fact, they can be used in an infinite variety of ways to aid in every step of the wet processing of textiles. And they are perfectly harmless to hide, hair and hoof. Not all the uses for these versatile oils can be listed here for it is likely that many dyers have developed particular applications for their own needs, applications which are not generally known.

Well Known Uses

Possibly the greatest quantity of Turkey Red Oil is usd directly; as a wetting, levelling, softening or finishing agent. This is the most common and familiar application and will not be dwelled upon here. Its advantages are ease in handling, easy and immediate solubility, resistance to hard water and the desirable finish imparted to textiles. Sulfonated oils are unaffected by and will work well in conjunction with the alkalies, salts and other chemicals ordinarily added to processing baths.

A most remarkable quality of sulfonated castor oil is its before mentioned ability to solubilize and emulsify practically all of the lubricants and solvents applied to textiles. Excellent yarn lubricants of the self-emulsifying type can be prepared with the sulfonated oils, and most every type of mineral oil from the very light fractions to the heavy-bodied engine oils may be incorporated. Some fatty acid is generally used to clarify and stabilize the mix. A high concentration of lubricant is obtained and yet the removal of these oils from fibers is very easy—warm water with perhaps a bit of soap and mild alkali is all that is necessary. Similar to the yarn lubricants are the soluble mineral oils applied to sulphur blacks and other shades for the finish and luster they impart. These are usually applied in the last rinse. An analogy to this, not generally known, is that many dry cleaners use a little mineral oil in their solvent to give luster to the clothing being cleaned. (Somehow it seems to me that most of this luster goes to the seat of my blue serge pants).

Water miscible wetting agents and solvents can be prepared with sulfonated oils in a manner hardly to be matched by any other single agent. Some of the penetrants commonly used today can be made by a simple mix of sulfonated castor oil and solvent. Many of the soluble pine compounds can be so prepared, and perhaps some of you prepare your own penetrant in this manner. However, it is often just as economical to buy a prepared penetrant because the manufacturer obtains his solvents in greater quantities at a saving which he is enabled to pass along in the finished product. Some percentage of practically every organic solvent can be incorporated in Turkey Red Oil by simple agitation, without impairing the clarity of the latter or of its solution. In general, a neutral finish fifty percent oil would be recommended. But most of the scouring and wetting agents on the market are not so simply prepared, for there is generally a high percentage of solvent and a definie pH to be maintained. Some of the products have a most delicate balance of sulfonated oil and solvent and any deviation either way will ruin both the clarity and the stability. Some times an additional agent is added as a mutual solvent—improving or making possible a clear homogeneous mix, or perhaps enabling the oil to "take" more solvent than would be otherwise possible. Ordinarily these compounds have a neutral finish, though in some instances a certain percentage of free fatty acid is desirable, and in other instances free alkali is advantageous. These factors are influenced by the nature of the mix and the specific use for which it is prepared. The finished product is nearly always a clear, viscous liquid, immediately and clearly miscible with water. It must be stable to temperature and to time, and maintain its original chemical and physical make-up. It must offer to the textile chemist and dyer the desirable properties of any particular solvent or penetrant in a soluble (that is, emulsifiable) form, ready for use without further compounding. The sulfonated oil and solvent compounds do this successfully and consistently. Other soluble solvent mixtures can be and are prepared, and give good results in use, but they seldom offer the ease of handling, speed of solution, ready availability and economy in operation that the former consistently deliver.

Little Known Uses

The above are some of the uses of sulfonated oils with which we are all familiar. However, there is a vast variety of uses not so generally known—not all of them important but all interesting in bearing out the central theme of the applications of these oils. Each of you, no doubt, will be familiar with some of the uses to be mentioned. It would be impossible to make this list complete, for I think that it can be truthfully said that no one person knows all of the applications to which sulfonated oils are put.

For the pasting of vat dyes, a neutral sulfonated castor oil is ideally adapted. The dyestuff is well mixed

with the oil, and caustic and hydrosulphite is then added. This gives the reduced dye, or leuco base, which then is in the proper state for application.

Turkey Red Oil is used in making up dye solutions for the spot dyeing or hand painting of satin slippers. The idea is of course applicable to any form or spot or daub dyeing and in fact there are other similar applications, both tentative and actual.

The softening properties of sulfonated oils are made use of in a great many ways. The oils have of course been used for years in softening all ordinary textiles, generally being applied in small portions in the last rinse. Sulfonated olive oil, sulfonated oleic acid, and sulfonated tallow find a heavy use as softening and finishing agents. They, as well as Turkey Red Oil, are often incorporated in various sizing, softening and fulling compounds. In connection with the softening effect of the soluble oils we also think of the finish which they impart—adding a soft lustre and a pleasant handle to the goods. To this we have already briefly referred.

In the manufacture of dental floss sulfonated castor or sulfonated olive oil is used to remove any scroop which the yarn may have, and to take out any possible harshness. Afterward, the floss is rinsed to remove any taste of the oil which might remain.

In obtaining moire effects on fabrics Turkey Red Oil is applicable and often used. In fact, it is an excellent aid in obtaining any novelty or specialty finish or feel.

Rather paradoxically, perhaps, (and yet illustrating again the versatility of the product) sulfonated castor oil is used in one type or method of delustering hose. Finely divided barium sulfate is pasted with the oil, and this paste is added to the bath. The fine powder is held in suspension and performs its function of delustering. Turkey Red Oil should be used with almost any type of delusterant, especially if there is a noticeable tendency toward streaking in the material. The oil helps to hold the dye level. Many dyers use sulfonated castor oil in their stripping baths to keep the goods in good condition and avoid the appearance and feel sometimes familiar to redyers.

Although it is not commonly known, excellent agents for the scouring of dyeing and finishing machines can be prepared, often by the dyer himself, with sulfonated oils. Any one or more of the grease dissolving chlorinated solvents, such as ethylene dichloride or dichlorethyl ether, is compounded with the oil by vigorous agitation and the mix is applied either directly or in hot water. The type and amount of solvent, and the method of application, must be varied to suit the conditions of use. If desirable, a little soda ash or other alkali may be used, in conjunction. In passing, it might be mentioned that sulfonated castor oil will clean paint brushes handily—and mixed with kerosene will remove grease and dirt from the hands. There is absolutely no harmful action on the skin.

In connection with the last sentence is the fact that the cosmetics industry is now consuming a considerable quantity of turkey red and other sulfonated oils. Many shampoo as well as permanent wave preparations are based on sulfonated castor oil. Brushless shaving creams also contain this versatile product. And there is one very attractively packaged compound which is to be added to the bath and is advertised to bring a breath of the pine woods to the bath room. Neutral Turkey Red Oil and a little of some aromatic pine derivative such as terpineol does the trick. Sulfonated castor oil is used to solubilize perfumes for the manufacture of theatre sprays, as well as other deodorants or antiodorants. In fact, water soluble perfumes for every sort of use are prepared by blending the essential oils with the sulfonated oils. A new wrinkle is that sulfonated castor oil is now put up in one gallon cans and sold by retail drug stores to the beauty parlor trade. Thus you see that Turkey Red Oil is used to beautify humans as well as textiles. One could speak at some length on the applications of sulfonated oils in the cosmetics and allied trades, but this is of only passing interest to us here. However, I want to mention that these uses were generally discovered or developed by the cosmetics manufacturers themselves, who needed a versatile and harmless emulsifying agent. I do not believe that their industry has ever been closely worked by any of the oil producers.

In the manufacture of pine oil, coal tar and creosote type disinfectants there are several types of emulsifiers used. Rosin soap is often recommended for the sake of economy. But disinfectants made with sulfonated oil as emulsifier usually show a higher coefficient (which is the measure of their effectiveness), for equal amounts of disinfecting agent, than do those made with soap and other emulsifiers. This is so because the sulfonated oil emulsions are more uniform, more stable, and give a much finer dispersion—this last factor is essential in a good self-emulsifying disinfectant. There is a considerable field open for the development of better insecticide and germicide agents through the use of sulfonated oils.

Sulfonated oils are used in preparing asbestos products. They are quite largely consumed in the processing of leather, and for this application sulfonated cod oil is often desired. Sizing preparations for paper and for book binding materials sometimes contain sulfonated castor oil. It is also used on wood in a few particular lines. Cutting oils for the metal workers can be and are made with sulfonated oil. And certain water soluble tints and stains are improved with soluble oils. And so on.

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It seems that I have wandered far afield in addressing a textile group. However, it has been a pleasure to illustrate a small measure of the versatility and the adaptability of the sulfonated oils—products which perhaps had been known to some of you only in their relation to certain special applications in the textile industry.

Mercerization of Cotton for Strength*†

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The mercerization of cotton yarn was studied at the request of the National Advisory Committee for Aeronautics. The object was to determine the conditions for the production of yarn having the maximum strength for a given weight. Experiments were conducted under strictly controlled conditions varying the tension on the yarn, time and temperature of mercerization, and concentration of caustic soda. Yarns varying from count 10 to 90, some of them in a series of twists, were mercerized.

The removal of all extraneous materials from the yarns by means of a thorough pretreatment was found to materially increase its strength. A further increase resulted when pretreated yarn having low twist, obtained with twist multipliers from 2.2 to 3.0, was mercerized. The maximum increase was obtained when sufficient tension was applied to the yarn in the caustic bath to prevent it from contracting more than 2 or 3 per cent, mercerizing at a temperature of 0° C. or lower, with a caustic soda solution having a concentration of 10 per cent or higher, for five minutes.

The results clearly indicate that the twist in the yarn must be low if the maximum increase in strength is to be obtained. The strength of such yarns can be increased from 40 per cent to 100 per cent over that of the original yarn of the same weight.

I. Introduction

THE process of mercerization is named after John Mercer who, in 1850, obtained a patent, one claim of which is that cotton fibers and cloth are strengthened by treatment with caustic soda or certain other substances. Another patent granted in 1890 to Arthur Lowe points out that the luster of cotton textiles is improved by mercerizing them under tension. From the time of Lowe's patent to the present, the primary object of commercial mercerization has been either to improve the appearance of yarns and fabrics by increasing their luster or to improve their dyeing qualities. However, there is a growing demand for cloth of the greatest strength for a given weight, especially for use in aviation. Hence the possibility, first pointed out by Mercer, of increasing the strength of yarn and cloth by mercerization is now of greater importance than at any previous time.

The literature indicates that the strength of cotton yarn or cloth can be materially increased by mercerization, but the conditions for obtaining the maximum increase in strength have not been worked out systematically.

The purpose of the study to be described was to determine the conditions for mercerizing cotton yarn to obtain the maximum strength for a given weight. Apparatus for controlling the variables was built and yarns were mercerized with it under systematically varied conditions of tension, time, temperature and concentration of caustic soda. Attention was given to the relation between the count, twist, and ply of yarns and the increase in strength resulting from mercerization.

The experiments were limited to yarn mercerization because of the better control that appeared to be possible. However, the results obtained should be applicable to the mercerization of cloth as well as yarn.

This work was undertaken at the request of and with the financial assistance of the National Advisory Committee for Aeronautics.

II. Materials and Equipment

Cotton yarn of count 80 spun from 1 5/16 inch staple length good middling Peeler cotton in the Bureau cotton mill was used for the study of the variables of the mercerization procedure. This yarn was spun with twist multiplier 3^1 giving about 27 turns per inch, slightly lower

*Publication approved by the Acting Director, Bureau of Standards, U. S. Department of Commerce.

†An abstract of this paper was read at the Annual Meeting in Greensboro, December, 1932.

Table 1

Results for the mercerization of yarns of different counts and twists. Caustic soda concentration 10%, temperature —10 degrees C., times 5 minutes, tension as indicated. The 10's and 20's yarn were made from 1 inch, the 35's from 1-1/16 inch, and the other yarns from 1-5/16 staple length Peeler cotton, unless otherwise indicated.

Nominal Count	Twist Multiplier	Ply Twist in turns per inch	ACTUAL COUNT						NOMINAL STRENGTH IN LBS. PER 100 ENDS						INCREASE IN STRENGTH OVER THAT OF GREY YARN IN PER CENT				
					Mercerized						Mercerized						Mercerized		
					Load during mercerization in lbs. per 100 ends						Load during mercerization in lbs. per 100 ends						Load during mercerization in lbs. per 100 ends		
			Grey	Pretreated	0.2	1.1	other	Double mercerization	Grey	Pretreated	0.2	1.1	other	Double mercerization	Pretreated	0.2	1.1	other	Double mercerization
90	2.6		84	94	87	94			18	22	21	25			22	17	39		
80	2.0		78	87	80	84			16	21	27	30			31	69	88		
80	2.2		80	83	81	85		77	17	24	27	31		29	41	59	82		71
80	2.5		77	78	69	79			20	26	21	31			30	5	55		
80	3.0		78	85	73	86			21	25	23	30			19	10	43		
80	3.5		74	78	72	78			22	25	23	27			14	5	23		
80	5.0		76	79	75	82			20	22	20	23			10	0	15		
80	6.7		70	82	81	79			13	15	16	16			15	23	23		
160 2-ply[a]			79	83	76	80			22	26	33	33		32	18	50	50		45
60	2.8		58	61	53		59[c]		32	35	33		40[c]		9	3		25[c]	
60[b]	3.7		61	67	56		62[c]		37	45	34		42[c]		21	— 8		14[c]	
40	2.4		41	43	39	44	45[c]	41	46	57	65	68	74[c]	69	24	41	48	61[c]	47
40	4.0		40	42	40	42	42[d]		48	52	54	57	54[d]		8	12	19	12[d]	
40	4.2		42	41	38		46[d]		58	63	47		63[d]		9	—19		9[d]	
80 2-ply	2.5	10	38	42	36	43		38	39	59	68	81		75	51	72	108		92
80 2-ply	2.8	18	40	46	40	44			53	63	70	76			19	32	43		
80 2-ply	2.9	30	36	38	36	38	41[c]		59	63	66	70	76[c]		7	12	19	29[c]	
80 2-ply	3.5	10	37	41	37	39	42[c]		52	63	66	75	77[c]		21	27	44	48[c]	
80 2-ply	5.0	25	39	41	37	42	43[c]		56	57	63	67	71[c]		2	12	20	27[c]	
80 2-ply	6.4	40	40	41	39		43[c]		44	50	50		59[c]		14	14		34[c]	
35	2.5		35	35	32		36[f]	32	23	43	54		58[f]	54	87	135		152[f]	135
35	3.3		32	37	32		36[f]		39	57	53		61[f]		46	36		56[f]	
35	4.5		34	35	33		37[f]		46	48	49		54[f]		4	7		17[f]	
20[d]	2.5		20	22	19	21[e]	19[g]	21	38	76	97	103[e]	105[g]	111	100	155	171[e]	176[g]	192
20	3.5		20	20	19		23[e]		78	94	102		116[e]		20	31		49	
20	4.7		20	20	19		21[e]		88	78	88		89[e]		—11	0		1[e]	
10	2.5		9.7	10.0	8.5	10.3[i]	9.5[i]	10	114	184	240	245[h]	245[i]	242	61	135	114[h]	115[i]	112
10	4.1		9.7	10.5	9.2		10.2[h]		163	170	172		188		4	5		15	
10	5.1		9.7	10.8	10.1		11.0		174	155	165		180[h]		—11	— 5		3[h]	

[a] Staple length and kind of cotton not known.
[b] Pima cotton.
[c] Load during mercerization 1.5 lbs. per 100 ends.
[d] Load during mercerization 2.25 lbs. per 100 ends.
[e] Load during mercerization 4.5 lbs. per 100 ends.
[f] Load during mercerization 2.6 lbs. per 100 ends.
[g] Load during mercerization 0.2 lbs. during rinse 4.5 lbs. per 100 ends.
[h] Load during mercerization 9 lbs. per 100 ends.
[i] Load during mercerization 0.2 lbs. during rinse 9 lbs. per 100 ends.

twist than is required to produce grey yarn of maximum strength. Yarns of count 80 made from the same cotton in other twists were used in studying the effect of twist. Information concerning certain other yarns used for the experiments is given in the title to Table I.

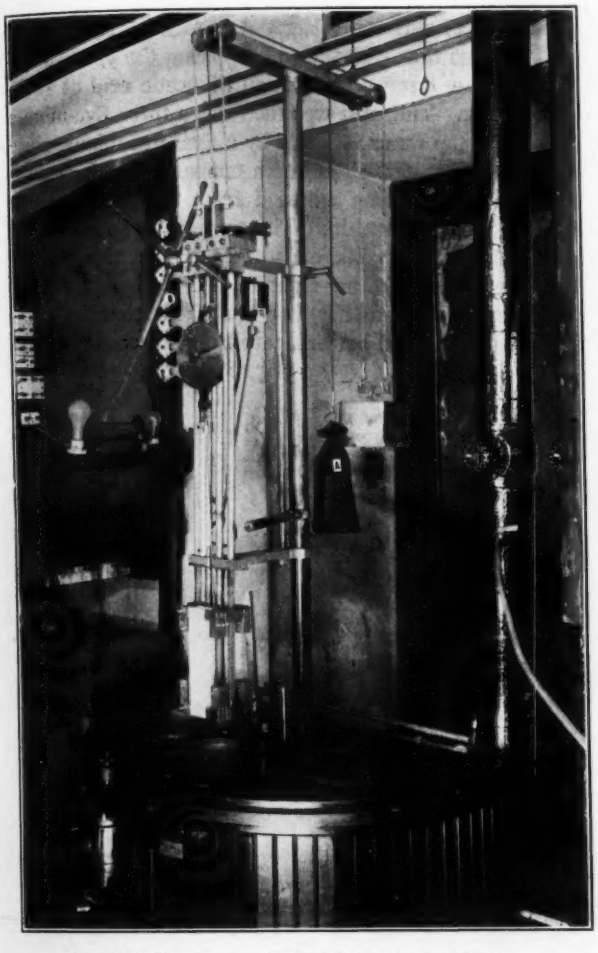

Fig. 1—Machine for mercerization of yarns in the laboratory.

Commercial caustic soda was purified for these experiments by allowing a 48.5 per cent stock solution to settle, the clear supernatant liquor being used. After this purification, the solution contained 0.001 per cent iron[2] and 0.5 per cent anhydrous sodium carbonate. This concentrated solution was stored in pyrex glass bottles which were equipped with ascarite tubes to prevent contamination from atmospheric carbon dioxide. It was diluted with carbon dioxide free distilled water for use in the experiments.

The apparatus which was designed for the mercerization of the yarns is shown in Figure 1. It consists of a constant temperature bath in which the containers for the solutions used can be placed and a mechanism for the support of the yarn which provides for the measurement and control of the tension on it. The mechanism is suspended as a unit by cables which pass over pulleys on the cross-tree at the top of the mast shown in the center of the figure to weights which serve as counter-poises. The mechanism can be lowered to bring the yarn into the solution below it. The mast can be rotated to bring the mechanism over any one of the three containers.

The bath is equipped with motor-driven agitators and is filled with water or, for low temperatures, with alcohol. It is provided with a coil through which either steam or cold brine can be circulated. An auxiliary electric heating unit controlled by a thermostat supplements hand control and permits the temperature to be held constant to within 0.5° for any temperature between about 96° C and —10° C. Three openings in the top of the bath (covered in the photograph) accommodate Monel metal containers of 15 liters capacity in which the caustic soda solution and rinse waters are placed.

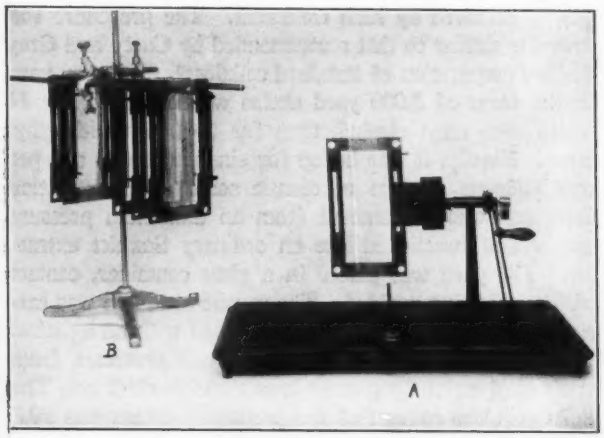

Fig. 2—Method of winding yarn on frames for mercerization.

The frames on which the yarn to be mercerized is wound and the winder are shown in Figure 2. The holes in the corners of the frames slip over studs on the supporting mechanism just above the bath in Figure 1 in which the six frames that are accommodated at one time are shown in place. The ends of the frames are held apart by detachable links (Figure 2) that are removed when the frames are placed on the studs (Figure 1). The lower studs are fixed. The upper studs are attached to a carriage that is free to move vertically on guide rods. The carriage is suspended by a cable from the spring scale which is attached above to a rack that is in turn suspended by a cable passing over pulleys and terminating in the bag of shot (A) in Figure 1. There is too much friction in this mechanism to permit the bag of shot to be

[4] Load during mercerization 2.25 lbs. per 100 ends.
[5] Load during mercerization 4.5 lbs. per 100 ends.

used as a constant known load on the yarn. It is simply used as a counter-poise. The load on the yarn is obtained by hand-operated pinion gears which engage the rack. The load on the yarn is indicated by the pointer on the spring scale. Another pointer, attached to the cable from the carriage to the scale, indicates on a ruler any changes in length of the yarn during processing. A propeller directly below the yarn driven by a small motor keeps the liquid in which the yarns are placed thoroughly agitated[3].

The apparatus used in preparing the yarn for mercerization is described in the next section.

III. Procedure

1. *Pretreatment*

All of the yarn used in the experiments was given the pretreatment now to be described. The purpose of the pretreatment was to remove cotton waxes, oils, and in fact all extraneous materials in order to prevent contamination of the mercerizing solution and to provide a uniform material for mercerization. A commercial preteatment was not given because foreign materials usually are not completely removed by such treatment. The procedure followed is similar to that recommended by Corey and Gray for the preparation of standard cellulose[4]. The grey yarn in the form of 3,000 yard skeins was extracted for 14 hours with ethyl alcohol, then for 14 hours with ethyl ether. Finally, it was boiled for six hours with a 1 per cent aqueous solution of caustic soda. The extraction apparatus was constructed from an aluminum pressure cooker and functioned like an ordinary Soxhlet extractor. The yarn was placed in a glass container, contact with metal being avoided. The caustic soda boil was carried out in a Monel metal can provided with an agitator. The yarn was kept submerged and was protected from mechanical action by placing it in a cheese cloth net. The alpha cellulose content of the pretreated cotton was 99.7 per cent.

2. *Mercerization*

The pretreated yarn to be mercerized was conditioned by exposure for at least 24 hours in an atmosphere of 65 per cent relative humidity having a temperature of 70° F. One hundred turns, about 56 yards, were then wound upon each frame with the winder shown at A in Figure 2, a very small but uniform tension being applied during winding. The frames were next placed in position on the apparatus that has been described. After the first frame was placed on the apparatus, the movable carriage was locked in position. The connecting links of the frame were next removed. The remaining five frames were placed in position in like manner, each having its connecting links removed. The carriage was then unlocked and the desired tension was applied to the yarn. This tension was maintained during mercerization, or changed as desired, by the hand-operated rack and pinion. The mechanism was then lowered to immerse the yarn in the caustic soda solution. After the caustic treatment was completed, the yarn was shifted over into another container filled with distilled water at a temperature of 0° C. or, if mercerization was carried out at room temperature or higher, 20° C. This rinse was followed by three five-minute rinses with tap water. Any caustic remaining in the yarn was then neutralized by placing the yarn for 10 minutes in a 1 per cent solution of acetic acid at room temperature. The yarn was then given three five-minute rinses with tap water followed by a final five-minute rinse with distilled water, all at room temperature. The mercerized yarn was then removed from the final rinse by elevating the mechanism. The tension was reduced to about 0.2 pound per 100 ends and the yarn was allowed to dry in a current of air from an electric fan.

When the yarn was dry, it was shifted from the metal frames to cardboard holders, care being taken not to dis-

Fig. 3—Mercerized yarn after transfer from the metal frames, 1 for strength tests, 2 for determination of count.

arrange it. Yarn to be used for the breaking strength test was obtained as shown at 1 in Figure 3. Gummed paper was pasted over the yarn to hold it to the cardboard. The yarn was then cut across at each end where it looped back, giving two sheets of strands. Yarn to be reeled and weighed for the determination of count was obtained as shown at 2 in Figure 3.

3. *Testing*

All tests were made after conditioning the yarn by exposure for at least 24 hours in an atmosphere of 65 percent relative humidity having a temperature of 70° F.

The count of the yarn, i.e., the number of 840 yard lengths in a pound, was obtained by calculation from the weight of a 45 yard length.

The breaking strength of the yarn was found as follows: The one hundred strands taken from one side of the frame as described above were inserted simultaneously (multiple strand method) in the jaws of a pendulum type tester. The capacity of the machine was 110 pounds except for testing the 10's yarn, when a machine of 300 pounds capacity was used. The distance between the jaws was four inches and the rate of reparation was 12 inches per minute. The elongation of the yarn at break was recorded by the machine. Six breaks were made for each mercerization and the results averaged.

Pretreatment or mercerization affects not only the strength but also the count of a yarn. Since the strength of a yarn is very nearly proportional to the reciprocal of the count for small changes in count, the strength of each yarn was multiplied by the ratio of its actual count to the nominal count of the grey yarn (given in the first column of Table I). The nominal strength thus obtained, that is, the (approximate) strength the yarn would have had if its count were the nominal count of the grey yarn, is a useful figure for comparison of the results of the treatments. It is the figure reported in this paper.

IV. Results

1. *Pretreatment*

Scouring, kier-boiling, bleaching or other wet treatments that may precede the mercerization of cotton yarn remove non-cellulosic materials and decrease the weight. At the same time the yarn may shrink or stretch. The very thorough pretreatment given the yarns used in this study resulted in changes in the nominal strength of from —11 per cent to 100 per cent of that of the grey yarn depending upon the size twist, ply and character of the yarn. In general, the low twist yarns showed the greater increase in strength. Data for a variety of yarns are given in Table 1.

The pretreatment also resulted in an increase in the diameter of the yarn, a fact of significance for aeronautical uses, for example in cotton parachute cloth, where the air permeability of the cloth must be controlled. The diameters of some of the yarns before and after pretreatment and mercerization are given in Table 2.

2. *Tension*

In order to determine the relation between the tension applied during mercerization and the strength of the resulting yarns, experiments were run in which the tension was varied and the other conditions were those reported to give the maximum swelling and optimum commercial result[5]. The caustic soda concentration was 23 per cent, the mercerization temperature was 20° C. and the time of immersion in the caustic five minutes. Three tensions were applied; 0.2 pound per 100 ends, which was the tension just necessary to eliminate the slack in the yarn, 1.1 pounds per 100 ends, and 2.8 pounds per 100 ends. In some experiments, the tension was applied to the yarn just before it was placed in the caustic soda bath and was not removed until after the final rinse. In others different tensions were used during immersion in the caustic and in the rinses.

Table 2

Effect of pretreatment and mercerization on diameter of yarn mercerized with a 10% solution of caustic soda, temperature —10° C., time 5 minutes.

Nominal Count*	Twist Multiplier of Yarn	Diameter in Millimeters			
		Grey	Pretreated	Mercerized Load During Mercerization in Pounds	
				0.2	1.1
80	2.0	0.134	0.135	0.100	0.094
80	2.2	.129	.124	.102	.086
80	2.5	.136	.134	.116	.110
80	3.0	.124	.132	.098	.091
80	3.5	.113	.121	.110	.093
80	5.0	.100	.105	.099	.092
80	6.7	.106	.112	.093	.102
40	2.4	.157	.163	.132	.130
40	4.0	.164	...	.151	.132
35	2.5	.200	.170	.143	.142
35	4.5	.148	.148	.146	.131
10	2.5	.435	.364	.343	.271
10	4.1	.318	.318	.309	.269

*For actual count and nominal strength see Table 1.

The results are given in Table 3. The strength of the pretreated yarn was not improved by mercerization. It was in fact decreased where the tension was only 0.2 pound. Later experiments were carried out under the conditions subsequently found to be more favorable to an increase in strength with yarns differing in size and construction. The results, which are given in Table 1, indicate that in general a tension greater than enough to eliminate slack is required for an increase in nominal strength over that of the pretreated yarn. Further, they show that, provided sufficient tension is applied, a considerable variation in the tension does not materially affect the results.

The last two experiments in Table 3 show the effect of applying tension to the yarn in rinse instead of during immersion in the caustic. They indicate that tension should be applied in the caustic bath.

The amount of contraction of the yarn in the caustic solution was observed. It was dependent upon the tension applied during mercerization. All of the yarns studied except those having relatively high twist contracted between 10 and 16 per cent when only 0.2 pound tension was applied to the yarn in the caustic solution. With the greater tensions tried, they contracted not over 3 per cent and some of them stretched 1 or 2 per cent. The yarns which contracted in the caustic all elongated somewhat during the rinsing.

The practical result of the contraction was the effect on the elongation of the mercerized yarn. The elongation at break of yarns mercerized under conditions permitting them to contract was equal to or several per cent higher than that of the grey or pretreated yarn whose elongation varied from 4 to 7 per cent (rarely 10 per cent). The elongation of the yarn mercerized under a tension of 1.1 pounds or more was usually from 2 to 5 per cent.

Table 3

Effect of tension on the strength and elongation of 80s yarn (Twist multiplier 3) mercerized with a 23% solution of caustic soda for 5 minutes at 20° C. Data for the grey and pretreated yarns are given for comparison.

(Loads given are per 100 ends of yarn)	Actual Count	Nominal Strength	Increase in strength over that of grey yarn in %	Elongation at break in %	Maximum Contraction in merceriza-tion bath in %	Contraction at end of final rinse in %*
Grey yarn	78	21	..	6	..	..
Pretreated yarn.	85	25	19	5	..	..
0.2 lb. in mercerization and rinses	73	21	0	10	15	11
1.1 lbs. in mercerization and rinses	83	25	19	2	0	—2
2.8 lbs. in mercerization and rinses	83	25	19	1	..	..
0.2 lb. in caustic and 1.1 lbs. in rinses	80	23	10	4	10	5
0.2 lb. in caustic and 2.8 lbs. in rinses	79	22	5	2	12	4

*Based on length of yarn before entering caustic solution.

3. *Time*

A series of experiments were performed in which the time of immersion in the caustic was varied from 30 seconds to one hour. A tension of 1.1 pounds per 100 ends and a 23 per cent solution of caustic soda were used in these experiments. The nominal strength of the resulting yarn remained practically unchanged as also did the elongation at the break. This indicates that for periods over 30 seconds the time of immersion under the specified conditions does not affect the strength of the yarn.

4. *Temperature*

The 80's yarn, twist multiplier 3, was mercerized under different conditions of temperature and concentration of caustic, the time of mercerization being 5 minutes and the load 1.1 pounds per 100 ends. The results are given in Table 4. The nominal strength of the yarn mercerized at 0 and —10° C. was somewhat higher than that of the pretreated yarn (nominal strength 25 pounds per 100 ends). Temperature appears to have little effect in the range 10° to 96° C.

5. *Concentration of Caustic*

Table 4 also gives the results of mercerization experiments covering a range of concentrations of caustic from 5 per cent to 48 per cent. There is no clear evidence that the nominal strength is materially influenced by variations in the concentration within this range. It was not possible to run an experiment with a 5 per cent solution of caustic at —10° C. because of the separation of a crystalline hydrate.

Table 4

Relation between temperature and concentration of caustic solution of the nominal strength of 80's yarn (Twist multiplier 3) when it was mercerized for 5 minutes under a load of 1.1 lbs. per 100 ends.

The values given are the nominal strength in pounds per 100 ends.

Temperature in degrees Centigrade	CONCENTRATION OF NaOH IN %						
	5	6	10	15	23	36	48
96	25	..	26	26	..	25	..
95	..	..	..	..	24	..	..
75	..	..	..	..	23	..	..
50	26	..	25	26	25	24	..
30	..	..	..	..	25	..	..
20	..	25	25	26	25	26	26
10	..	..	..	..	27	..	26
0	..	26	..	..	28	26	..
—10	..	..	30	29	28	..	..

6. *Twist*

The results of the experiments already described led to the selection of the following conditions for a study of relation of yarn twist to mercerization for strength; tension 1.1 pounds per 100 ends for 80's, time five minutes, temperature —10° C., caustic concentration 10 per cent. A series of 80's yarns spun from the same cotton with twist multipliers ranging from 2 to 6.7 was mercerized under these conditions. Yarns of other counts, some of them in several twists, were mercerized under the same conditions except that the tension applied to the stronger yarns was greater. The results are given in Table 1. Some of them are presented graphically in Figures 4 and 5. The pretreatment of the grey yarn increased the strength of the yarns of twist multipliers up to 3.0 much more than that of the higher twist multipliers. In some cases it actually weakened the yarns having higher twists. Mercerization under a tension of only 0.2 pound per 100 ends usually increased the strength of low twist yarns over that of the pretreated. In some cases it decreased the strength of the higher twist yarns. In every instance when a greater tension was applied, mercerization resulted in

yarn having greater nominal strength than either the grey yarn or the pretreated yarn. The maximum increase over the strength of the grey yarns was in excess of 150 per cent for the low twist, low count yarns and 80 per cent for the low twist 80's.

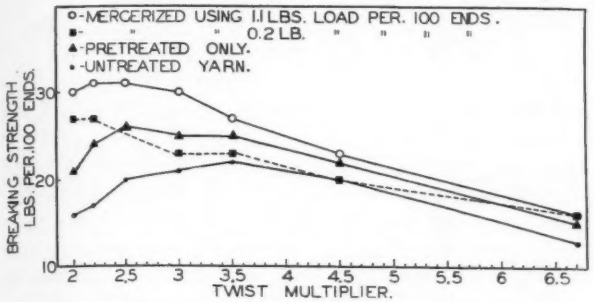

Fig. 4—Relation between breaking strength and twist, 80's cotton yarn.

Two-ply 160's and two-ply 80s' yarn were mercerized with results similar to those obtained with singles of the equivalent count.

8. *Double Mercerization*

From the results given above it appeared that a tension greater than 0.2 pound per 100 ends applied while the yarn is in the caustic solution is essential for the maximum increase in strength. The elongation at break of yarn so mercerized is very low, usually about 2.5 per cent and practically never exceeding 5 per cent. Yarn with too little stretch is undesirable for many purposes.

Some experiments were carried out in which the yarn mercerized under conditions giving the greatest increase in strength was mercerized again with less tension. The second mercerization differed from the first in that about 15 minutes were required for complete contraction of the yarn in the caustic, whereas the contraction in yarn being mercerized for the first time was complete within two or three minutes. This double mercerization resulted in yarns having the maximum strength obtainable in a single mercerization and also any desired elongation up to about 10 per cent, depending upon the amount of tension used in the second mercerization. Results of double mercerization experiments are given in Table 1.

It appeared that yarn having the maximum strength but greater elongation might be obtained by other procedures than double mercerization. Thus the yarn could be mercerized under tension in the usual way and the tension then decreased before the yarn is removed from the caustic in order to allow it to contract. Several such procedures were tried. Although it was possible to increase the elongation in this way, the strength was not always equal to that obtained by the double mercerization procedure.

V. Discussion of Results

The pretreatment given the yarn for these experiments was more thorough than the usual commercial pretreatments, mercerization was carried out with an extremely large volume of fresh, clean caustic solution, and the rinses were very thorough. Consequently, the increases in strength obtained are for what may be considered ideal rather than practical commercial conditions. However, they can be used as a measure of the efficiency of commercial processing.

The pretreatment itself resulted in a very considerable increase in the nominal strength of all yarns of low or medium twist. Whether this pretreatment is essential for the increase obtained by combined pretreatment and mercerization was not determined.

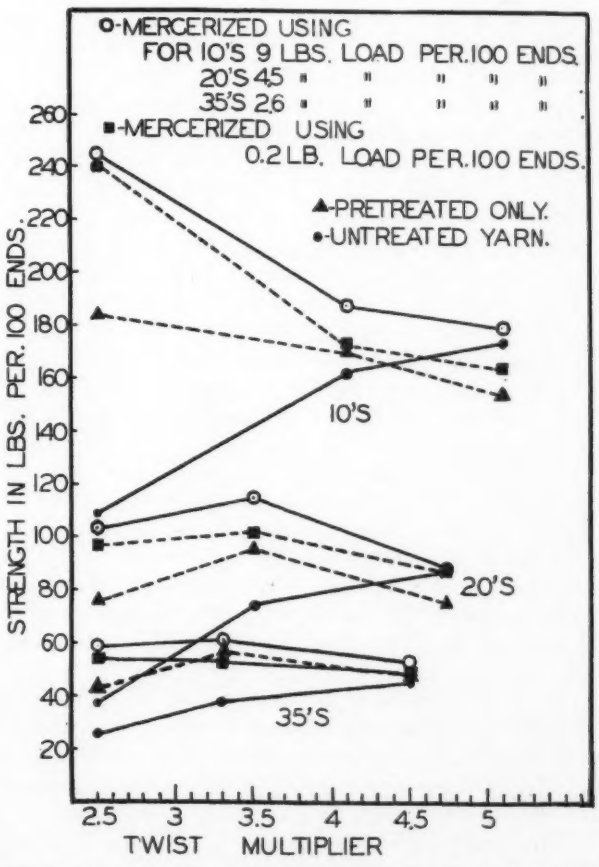

Fig. 5—Relation between breaking strength and twist, 10's, 20's, and 35's cotton yarn.

Because of their greater diameter, the pretreated yarns may be of more value for some uses than the mercerized yarns even though the latter are stronger.

The strength of a thoroughly pretreated yarn can be still further increased by suitable mercerization. Unless

the twist of the yarn is low, tension of the order of 1 or 2 pounds per 100 ends during mercerization is necessary to obtain this increase and even in the case of the low twist yarns, the increase is greater when tension sufficient to prevent the yarn contracting more than 2 or 3 per cent in the caustic bath is used. The time of mercerization, temperature, and concentration of caustic need not be carefully controlled, though somewhat greater strength results when the caustic concentration is low, say 10 per cent, and the temperature is kept at 0° C. or even lower.

The results clearly indicate that the twist in the yarn must be low if the maximum increase in nominal strength is to result from mercerization, and if yarns of maximum nominal strength are to be obtained. This effect of twist is so great that an 80's yarn of twist multiplier 2.2 having a strength of only 17 pounds per 100 ends is stronger after pretreatment and mercerization than a similar yarn of twist multiplier 3.0 having an initial strength of 21 pounds per 100 ends. Similarly for plied yarns, where the singles and ply twist are low the increase in strength resulting from mercerization is greater than when the twists are higher. In the case of 80's two-ply yarns differing only in twist, the strength of the yarn having low singles and ply twist was doubled as a result of pretreatment and mercerization.

Some reasons for the increase in strength resulting from pretreatment and mercerization may be suggested. It is generally recognized that grey yarns having low twist are weak because of fiber slippage and yarns having high twist are weak because of torsional stresses on the individual fibers. An intermediate twist permits less slippage yet does not introduce undue torsional stresses and therefore results in yarn of maximum strength. This is illustrated by the curve for the untreated yarn in Figure 4.

Some of the factors which probably contribute to the increased strength of the pretreated and mercerized yarns are increased friction between the fibers, increased strength of the fibers, and better distribution of the load among them.

It is recognized that a better distribution of the load among the fibers may take place during pretreatment and especially during mercerization when the fibers are in a plastic condition, but the extent to which this contributes to the increase in strength is not evident.

The removal during pretreatment of material which lubricates the movement of fiber on fiber or which does not contribute as much to the strength of the fiber as the same weight of cotton cellulose, would increase the friction between fibers and increase the ratio of their strength to their weight. The data in Tables 1 and 2 clearly show that the count and diameter of the pretreated yarns of lower twists are greater than the count and diameter of the corresponding grey yarn, that is, the weight per unit length is less and the diameter more. The pretreated yarns feel harsher than the grey indicating an increase in coefficient of friction of the fibers and an examination of the broken ends of the yarns shows that there is less fiber slippage when they are broken than when the grey yarns are broken. These facts indicate that increased friction between the fibers is probably the predominant reason for the increased strength of pretreated yarns.

The low twist yarns mercerized under a tension of 0.2 pound have both smaller diameter and lower count than the corresponding pretreated yarns. (Tables 1 and 2.) This means that the fibers are in closer contact and the area of contact between them has increased. The broken ends of these yarns indicate the absence of fiber slippage. Just as the increase in strength of low twist grey yarns with twist is attributable to decreased fiber slippage, the increased strength of these yarns may be attributed to decreased fiber slippage. The yarns having higher twist did not change much in diameter and count when mercerized under 0.2 pound tension. It is not surprising to note that their strength after mercerization differs but little from that before, since no fiber slippage is observed in either case.

When the yarn were mercerized under the higher tensions they decreased but slightly more in diameter than when mercerized under 0.2 pound tension and changed but little more in count. No evidence of fiber slippage was found by examining the broken ends. However, the strength was in general greater than that of the yarns mercerized under the 0.2 pound tension. The increase would appear to be attributable to an actual increase in fiber strength. The study of how mercerization would increase the strength of the cotton hair is beyond the scope of this paper.

VI. Conclusion

The general conclusion to be drawn from this work is that the strongest mercerized yarn of a given count from a given quality of cotton is obtained under the following conditions:

1. Use of low twist yarn obtained with twist multipliers from 2.2 to 3.
2. Thorough pretreatment of the yarn to remove all extraneous materials.
3. Mercerization at a temperature of 0° C., or lower.
4. Use of sufficient tension during mercerization to prevent the yarn from contracting more than 3 per cent.
5. Use of caustic solution having a concentration of 10 per cent or higher.
6. The time of mercerization to be five minutes.

The resulting yarn should be from 40 to 100 per cent stronger than the original yarn of the same weight.

VII. Acknowledgement

The author is indebted to William D. Appel and Herbert F. Schiefer for assistance in interpreting the results and preparing this report.

References

[1] The twist multiplier (M) is a constant relating the number of turns per inch (T) given the yarn in spinning to the yarn count (C) according to the equation $T = M \sqrt{C}$.

[2] Unpublished work by the author shows that cotton is appreciably degraded, i.e., the alpha cellulose content is decreased, by mercerization at 100° C. with caustic soda solution containing only 0.01% of iron but that it is not degraded when the iron concentration is less than this amount.

[3] The apparatus is more elaborate than would be necessary if it were designed only for the present work. It can be used for the mercerization of skeins as well as of yarn wound on frames.

[4] A. B. Corey and H. LeB. Gray. Notes on the preparation of standard cellulose. Eastman Kodak Co., Rochester, N. Y. Ind. & Eng. Chem. *16*, pp. 853, 1130, 1924.

[5] Skinkle, J. H. Present Practice in Warp Mercerization. Textile World *75*, pp. 2377-2378-2427, 1929.

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A FUNDAMENTAL STUDY OF VAT PRINTING PASTES

A research project made possible through the support of the Textile Foundation.

THE Research Committee of the A. A. T. C. C. announces that preliminary plans have been made for a new research project, entitled "A Fundamental Study of Vat Printing Pastes," which will be started at once and is likely to arouse considerable interest in textile printing circles.

Among the numerous investigations which it is hoped to include in this research are the following:

1. An examination of representative vat printing pastes, with the object of accurately measuring their viscosity at different temperatures, resistance to shear, and other characteristics of a physical nature on which their successful working depends, so that it will be possible to define in mathematical terms those properties which make an ideal printing paste.

2. A study of all the thickeners which may be used in vat printing, including the proper methods of preparing them, their behavior in the presence of alkalies, hydrosulfite and other chemicals employed in vat printing pastes, and their relative efficiency from the standpoint of color value, fastness, penetration, sharpness of outline, stability, etc.

3. A study of the effect of different alkalies, catalysts, stabilizers, hygroscopic and dispersing agents, etc.

4. An accurate determination of the ratio between the total amount of dye printed and the amount actually fixed on the fiber, on various types of fabric.

An Advisory Committee, consisting of William H. Cady, Chairman, Arthur E. Hirst, Alfred L. Lustig, Morell Mackenzie, Louis A. Olney and Robert E. Rose, has been appointed to have general supervision of the project, and Silvert N. Glarum, a graduate student in the Department of Chemistry of Brown University, has been engaged as Research Associate to conduct the investigations. It is planned to have the more strictly scientific phases of the work carried on in the University laboratories, and the practical experiments involving the use of textile machinery will be performed in the plants of the U. S. Finishing Company and at the Lowell Textile Institute.

The research is being financed by funds provided by the Textile Foundation, and is sponsored by the A. A. T. C. C.

While the project is primarily limited to vat dyes, it is possible that some consideration may be given to the insoluble azo dyes in their various forms (Fast Color Salts, Rapid Fast and Rapidogen dyes) since they are so closely identified with the vat dyes in the production of fast colors.

The Advisory Committee would welcome any suggestions for the further extension of this investigation, so that it may cover the ground as completely as possible. References to previous researches, public or private, along similar lines would be particularly appreciated. Communications should be addressed to William H. Cady, U. S. Finishing Co., Providence, R. I.

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BANKS AND RESEARCH

NOT having in the past connected our banking institutions with research, it is with interest that we read the remarks of Mr. J. Beaumont Pease in the February 13th issue of the *Dyer,* an English contemporary publication. Mr. Pease is the chairman of Lloyd's Bank and in the course of his speech to the shareholders on February 3rd said in part:

"But though a prime condition of industrial recovery is, of course, the solution of the difficult financial problems of an international character with which we are confronted today, that in itself will not ensure the return of prosperity to this country, unless we exercise the same resourcefulness and enterprise in face of the circumstances of today which our predecessors showed in no less difficult though somewhat different circumstances in the past.

"It is perhaps true that the lines of development which were open to them are not the same as those which present themselves to us. Countries, which in their day were largely undeveloped, are now thriving communities, having industries which are offering serious competition. Today, there is probably no direction affording a better prospect of rich reward than scientific discovery. It has to be remembered that the exercise of the native and untutored genius for improvization and invention which, in the past, was sufficient to keep us in the van of progress is no longer sufficient. Economic production is today a highly expert business requiring the specialized training such as is possessed by the man of science. We must therefore turn more and more to the research worker to point the way towards new advances and industry must realize that an efficient and well-equipped research organization is an essential element of good management.

"Some of our industries provide notable examples of what can be done in this way, but facilities for industrial research are open to all, and there are numerous Government establishments, trade research organizations, as well as university institutions, to which an industrial firm may turn for help in the solution of its problems. If it is argued that this involves expense and that the necessary finance is not available, except for the larger and richer firms, I agree that some organization for providing finance for intermediate loans of modest amounts, and for comparatively short periods, which would fill the gap between ordinary banking loans and those of a longer duration and for larger sums, would be a desirable addition to our financial machinery. Steps for the formation of such an organization should be, and I may say, are being, taken."

In commenting upon this the *Dyer* goes on to state:

"This scheme seems to be a promising beginning and we look forward to the day when the banks will as a matter of course, *give* substantial sums to research organizations. After all, the banks derive a considerable proportion of their profits from industry and it is just as much their interest to support the research institutions as it is that of the actual manufacturer."

To date we have not read nor heard of the attitude of American banking institutions in regard to research. Undoubtedly they have an indirect interest in research developments but to what extent this interest prompts them to give actual support to research associations we do not know. It is encouraging to read a statement coming from a banker who is a convinced believer in the necessity for industrial research and the incurring necessity for the proper financing of such research.

We hear many reports concerning our banks and their doings and many bitter statements have been made concerning them. They have been described as unfit to carry on traditional American institutions and so on. We believe that President Roosevelt's recent reorganization drive will stifle such comments to a marked degree in the future. However, we are firm believers that our industrial future depends to a large extent upon proper scientific research, adequately financed. In many cases our banks could be of material assistance in this necessary financing. We have the proper men to carry on this work, we have the demand for it, and we have the organizations to properly direct this work. We shall await with interest a statement from our banking institutions regarding their attitude in this direction.

Textile Foundation News

Light, Heat and Textiles

Both retailer and housewife are seriously affected by the deteriorating effect of light, especially sunlight, on piece goods, dresses and other textile products. Shop window displays, household drapes and other exposed textiles are common prey to the destructive influence of light and heat. Considerable attention is being focussed, therefore, on the practical research toward the prevention or minimizing of that condition now being sponsored by the Textile Foundation.

Not only the appearance but also the quality of textile fabrics is jeopardized by exposure to light and heat, states Textile Foundation Research Fellow D. J. Salley, who is investigating the problem at Frick Chemical Laboratory, Princeton University. A 10-degree increase of temperature may double the rate of deterioration, Mr. Salley explains in a recent report to Franklin W. Hobbs, Chairman of the Board of Directors of the Foundation. The ultra-violet rays of sunlight are also particularly devastating, he states.

In addition to studying the exact action on the cellulose structure of textiles by light and heat, Mr. Salley's research is planned to discover the influence of various foreign substances added in textile manufacture that may hasten the rate of deterioration. The exact influence of such materials will be demonstrated for the practical purpose of enabling manufacturers or processors to prevent or minimize the condition where possible.

Another angle of the investigation, Mr. Salley explains, is to determine whether other substances may be employed to counteract the destructive effect of light and heat. He cites the instance of rubber, in which "anti-oxidants" are incorporated to combat the natural deterioration of aging.

Mr. Salley's report adds: "Chemical processes accelerated by light fall in a class known as photochemical reactions. The deterioration of textiles on exposure to sunlight clearly illustrates that condition.

"Some photochemical reactions result from light arousing only one particular molecule, but in other cases the light energy stirs a number of different molecules. These latter cases, called "chain reactions," are sometimes caused or aggravated by the presence of added substances which act as catalysts. Traces of iron or copper salt, for example, accelerate reactions in many oxidation processes.

"Conversely, the presence of small amounts of different foreign substances, usually some organic compound, may sometimes greatly decrease the rate of reaction. Such inhibitors serve to break the reaction chain at some stage. The use of "anti-oxidants" to forestall the aging of rubber is a case in point.

"In order to devise a method to prevent or minimize textile deterioration from photochemical reaction, our research will seek to ascertain the exact course of the oxidation process. The problem is particularly complicated because of the complex structure of the cellulose molecule which goes to make up the textile fiber. Simpler substances, related to cellulose, are being investigated first so that the manner of their exidation may be applied in the search for a control of textile oxidation."

Control of Dye Production

Textile dyeing is commonly regarded as secondary to textile manufacturing, but the style value accruing from beautifully dyed or printed fabrics has frequently been the leading factor in stimulating textile sales, according to Frank D. Cheney, Director of the Textile Foundation. Because of the close, mutual dependence of these two industries, Mr. Cheney states, the Foundation is supporting research to promote economies through more accurate control of dye production.

"Dyestuffs production revolves around the so-called 'benzene ring,' " it is explained by John S. Reese, Textile Foundation Research Fellow, who is working on the problem under Dr. Lapworth, Professor of Chemistry, University of Manchester, England. "Benzene occupies its fundamental position in the dyestuff industry because of its distinct and almost indestructible characteristics," Mr. Reese writes in a report to Director Cheney.

"Benzene is frequently found doubled or tripled within itself. Furthermore it can easily take on new parts and thus be almost infinitely varied without losing its characteristic property. The benzene ring of six fundamental parts is pictured by the chemist as a hexagon. At each of the six points there is opportunity for variation through the addition of new parts or substituents.

"Rather oddly, these substituents, when added to the benzene ring, do not choose any of the six points at random but only certain ones, depending among other reasons on the nature of the substituent which first locates itself. Efforts to describe the phenomenon of the orderly manner in which these new parts orient themselves have created the problem of the Orienting Influences of the Benzene Ring.

(Continued on page 235)

Washington News Letter

(Continued from page 206)

and other investigations which can be conducted effectively only by the Government would be continued under the same restrictions as technologic investigations.

It is proposed that as a matter of economy in Federal expenditures and also to test the seriousness of the desires of those seeking the continuance of industrial research for the benefit of industry, there be consideration of the adoption of the principle of reimbursement of the U. S. Tresaury by the interests directly served. Finally, this school of thought has advanced the premise that it is the duty of Congress to take under advisement the placing of restrictions on cooperative studies paid for by outside agencies if a new form of competition, as objectionable to the conservatives as the ones above mentioned, is not to be allowed to develop. By way of beginning, the foes of paternalism urge that Congress keep a sharp eye upon the present policies of all technical bureaus accepting cooperative research funds to the end that joint investigative work be continued "on a proper plane."

A Concession for Textile Design Applications

Following the steps recently taken at the Patent Office to speed up the examination of textile designs and the issuance of patents thereon, the administration has undertaken a twin project in behalf of the textile industry which has as its objective the reduction of the expense of patenting fabric designs. Under the old conditions it was necessary to file a drawing of the design with a design patent application. According to statements made to the officials by applicants, an expenditure of from $50 to $75 was sometimes necessary to obtain a suitable drawing. By virtue of an order lately issued by Commissioner Thomas E. Robertson, this part of the expense of design protection has been reduced to a minimum outlay of $3 to $5.

The gist of the modification of Regulations is that the Patent Office declares itself henceforth willing to accept, in lieu of India ink drawings,—*photographs* (not photostats) of designs in textile design applications, provided such photographs are printed on approved stock of the standard size and that the prints are so made, properly developed, fixed and thoroughly washed that they will not fade. In the beginning, Bristol board was specified for the photographs but it was later found that appropriate matt stock is equally effective or even superior for the purpose. In any event, the design photographs to be acceptable at the Patent Office must have blacks that appear as pure black on the prints and whites that are pure white. The middle tones must be interpreted by sharp dots of intense black.

In the case of shaded color designs, the new Regulations at the Patent Office prescribe a special process for making the prints. A negative is to be made on a panchromatic dry plate through a suitable filter to bring out the various shades in the design. From this negative, a halftone screen positive is made by the wet plate process, the ruling of the screen to be 65 lines per inch. From this positive a negative is made in film by contact printing in a vacuum frame from which negative the finished print is made on approved board or paper stock. Since the Patent Office has, by its Order, evidenced a disposition to let down the bars for cheaper equivalents of textile design pen-and-ink drawings, various alternatives of the straight photographic medium have been put forward in the trade and certain of these have been approved and accepted at the Patent Office. Particularly successful in this respect are certain utilizations of photolithography for textile design reproduction.

The Color Standardization Budget

The Director of the National Bureau of Standards has made a strong plea to Congress for a repetition, next year, of the allotment of about $12,500 necessary to continue the color standardization work. He has pointed out that the development and maintenance of a system of color standards serves a surprisingly wide variety of trade activities, including edible oils, paints, enamels, dyes, illuminants, railway and traffic signals, etc. He added that an increasing demand was anticipated for testing Lovibond glasses. He commented on the fact that in the Bureau's set of 465 glasses the grades of the red glasses have been carefully standardized and that a similar standardization should now be made for the yellow and blue glasses so that the Bureau may accept all kinds of Lovibond glasses for testing.

The investigation of textiles represents another activity for which the National Bureau is seeking a sustained appropriation,—some $50,000 a year in this instance. To indicate the practical consequences of the textile work, the Director of the Bureau has been telling Congressmen of the recent experiments which indicate that it may be possible to cement cotton fibers together into a strand without spinning, the result being a fabric not only possessed of greater strength but characterized by the appearance and feel of linen,—qualities that have been retained, in the tests at the Bureau, even after repeated laundering.

Rayon Dyeing and Finishing

(Continued from page 204)

Production of Cuprammonium Rayon

The cuprammonium process is the only commercially successful rayon process that depends upon the direct solution and precipitation of cellulose. The cuprammonium process depends upon the solubility of cotton in an ammoniacal solution of cupric copper and the subsequent precipitation of the dissolved cellulose in a setting bath.

Cellulose is treated more mildly in the cuprammonium process than in the other process of rayon manufacture. This fact would have greatly increased the use of the cuprammonium process were it not for certain nullifying factors. The first of these is the high cost of the process due to the use of large quantities of ammonia and copper, an appreciable quantity of which is lost during the process. Another factor affecting the growth of the cuprammonium process is the fact that the cost of the process makes it necessary to produce multi-filament yarn in order to successfully compete with cheaper processes. Multi-filament yarn made by any process must be expected to give more trouble during the textile operations than does yarn of relatively the same strength manufactured with coarser and therefore stronger individual filaments. The difficulty of successfully manufacturing rayon fabric from multiple filament rayon yarns on machines designed to handle coarser and stronger yarns, has proven so great that the growth of the cuprammonium process has been greatly retarded.

A third factor is.—the physical properties of cuprammonium rayon vary somewhat from the physical properties of the other rayons, particularly with respect to elongation.

Purified cotton linters are added to a solution of copper in concentrated ammonia, that is;—finely divided copper is dissolved in concentrated ammonia until the necessary concentration is obtained and to this is added a measured quantity of pure cotton. The cuprammonium spinning solution thus prepared is carefully filtered and stored until required for the spinning process.

The spinning solution is forced through a succession of filters into individual spinnerettes from which it merges into the setting bath. The setting bath is purposely made up in such a concentration that the cellulose is precipitated slowly rather than almost instantaneously, as in the viscose process. This slow coagulation allows the yarn to be stretched tremendously during spinning, thus enabling multi-filament yarn relatively free from broken filaments, to be successfully manufactured.

Immediately after the spinning operation, the yarn is rinsed free from all but minute traces of the copper with cold dilute sulfuric acid. Following the acid rinse, the yarns are thoroughly rinsed, neutralized and finished in a soap and oil solution, extracted and dried.

The dried cuprammonium process yarn is then wound into the form of "put-up" desired by the trade, and is stored pending shipment. At the present writing, an appreciable quantity of this yarn is sold in an untwisted condition in response to a specialized demand for such yarn, by certain branches of the knitting industry.

Constant Improvement in the Rayon Industry

Those who have had occasion to handle rayon during the past few years are well aware of the constant improvement that has taken place in the finished product. The rayon of today is vastly superior in appearance, uniformity and strength to the rayon of three years ago. The research staffs of the various companies will continue to find ways and means of improving the present product. Each of these improvements will temporarily complicate matters for the manufacturers of rayon fabric, but will ultimately react decidedly to the benefit of all concerned.

Probable Development of New Processes

In the foregoing pages, we have briefly discussed four methods of rayon manufacture that have achieved commercial success. On a laboratory scale artificial silk fibers have been manufactured by many other processes. To date none of these has been commercially successful because of the economic problem involved. The advantages of these non-commercial processes are not sufficiently great to offset the increased cost of installation and manufacture.

At the present time a considerable amount of work is being done on the production of rayon by processes quite different from those now in general use. It is very probable that some of these processes will achieve commercial success, and will result in the production of rayon having properties decidedly superior to the rayons now on the market. Obviously, it is impossible to foretell which of these processes may eventually achieve success, and which will present economic problems that cannot be overcome.

(To be continued in next issue.)

Processing of Thrown Silk

(Contributed by the Laboratories of Jacques Wolf & Co.)

Silk throwing is the term applied to the several operations by which the raw silk thread is converted into yarn for the knitting or weaving trade. Both raw and thrown silk are bought and sold on specifications called "Raw Silk Rules," which were devised by the Silk Association of America. These rules govern transactions between buyers and sellers in the United States of America and cover contracts, deliveries, weights, variation of size, seuplaire inspection, and terms of sale.

Because of the hygroscopic qualities of raw silk and the uneven moisture conditions of the shipping points in the Orient, all raw silk is purchased on "Conditioned weight" which is the absolute dry weight plus 11%. Thrown silk is sold on a "Conditioned Clean Fibre" weight plus a regain of 11%. The United States Testing Company is the official conditioning house of the American Silk Association.

The term "Clearance" is of vital importance to the buyer, manufacturer and seller of thrown silk. Clearance means the difference between the invoice weight of raw silk received and the weight of thrown silk delivered. It is sometimes erroneously believed to be the difference between the conditioned weight received and the thrown weight delivered and this interpretation has brought about very large clearances, some as high as 8% and 9% over the invoice weight. This excess clearance has brought an added task, and expense to the throwster, and the dyer, since the throwster must use more soaking material and the dyer consumes more time in stripping, and often the stylist of the fabric receives a gross disappointment. High clearances mean an excess boil-off and when this yarn is woven the space which should have been occupied by silk is filled up by soaking material. A high boil-off also means short yardage. Some very interesting work was done with yarns of various boil-offs by Mr. Irving Lewin of Duplan Silk Corporation and were published a few years ago. His table showed that a crepe yarn having the correct conditioned weight and a 25% boil-off gave the best yardage and best finished cloth.

Confidence in the throwster to return a legitimate amount of thrown silk with a 25% boil-off and constant yardage would certainly prove a boon to the silk industry in general.

We have recently developed an oil which requires only a small quantity to give a 25% boil-off and a legitimate clearance; it has proven very successful on six and eight thread 20/22 denier yarns and it leaves the silk immediately when it comes in contact with the boil-off bath, aiding that operation by acting as a wetting-out assistant.

On account of the unequal importance of the different cleanness defects in the manufacturing and finishing processes and in their effect upon the quality of the finished goods, a set of inspection rules must be carefully followed by silk inspectors who are trained in that particular field.

The sizes of raw silk used in the weaving trade are 13/15 denier or an average of 14 deniers and 20/22 deniers or an average of 21 deniers. The yards per pound of a one denier silk is 4,464,528 and by dividing this figure by the average denier size any yardage can be determined.

The foregoing gives some idea about the sale and purchase of the "Queen of Textiles" and shows how specialized that business is.

The throwing proper consists of crepe, tram or organzine twist; crepe is thrown silk having 20 to 100 turns per inch, more or less for use in making crepes such as georgettes; tram is a number of strands of raw silk soaked, doubled and twisted into a single thread; organzine is a kind of thrown silk twisted like a rope with strands to increase its strength.

The various processes of crepe yarn are:

1. Soaking.
2. Conditioning.
3. Winding.
4. First time spinning.
5. Second time spinning.
6. Setting of twist.
7. Redrawing.
8. Dulling or copping.

The raw silk as received in bales from the Orient is dry and brittle. To facilitate the various throwing operations, and improve the quality of the ultimate finished fabric, it is necessary to soften and lubricate the fibers and the outer coating of gum or sericin. Duirng the last year a considerable amount of research work has been carried on for the purpose of determining the basic principles in regard to the soaking process and its effect upon the finished fabrics. The soluble oils used as a base for these soaking oils must be from pure non-drying animal or vegetable fats and sulfonation controlled absolutely to avoid dry yarns, oxidation, and rancidity. The blended oil must be so balanced that the throwster gets maximum efficiency of take-up and money value which in turn has a direct bearing on the thrown clearance. In order to get permanent softness and lubrication these oils must penetrate the fiber interstices and not just coat the outside surface of the gum.

The soaking operation consists of immersing the skeins into a bath of soluble oil and soft water at a temperature

of 85-95° F. for twelve hours, this method is called tub soaking. Several ingenious machines are also in use which do the soaking in from one-half to two hours, thereby decreasing the inventory investment in the soaking department considerably. After soaking, the skeins are removed from the tub with care and hydro-extracted, after which they are sent to the winding department.

Conditioning or seasoning to allow the soaked skeins to partially dry out is the next process. This process requires three to four hours, before placing on the swift to be wound in order to avoid stretched yarn.

Winding is the process of transferring the soaked, conditioned silk from skein to bobbin or spool. It usually takes about four hours to wind a skein of Japan silk onto a bobbin. The tension on the swift must be carefully predetermined to avoid stretched silk.

First time spinning is really a doubling process where a small amount of twist is put into the several ends as they are drawn into one single continuous thread. The number of ends drawn into one gives the number of the thread such as six thread means six bobbins were drawn into one thread. The spindle speed of this operation varies from 4,000-6,500 according to the weight of endage, whether four thread or 12 thread.

Second time spinning is the operation in which the finished number of turns per inch are put in. The bobbins from the first time operation are placed on the supply spindles and spun upward to a much slower speed than the supply which varies from 9,500 to 12,000 r.p.m.

The full take-up bobbins from this latter spinning are now placed in a steam chamber where the yarn is set or made lifeless on the bobbin. Next to soaking, this steaming is very important since the time, temperature, moisture, and position of the bobbins play a part which will mean success or failure of the yarn.

After the bobbins of twist set silk have dried sufficiently, they are redrawn through cleaners to take out waste and bunches and at the same time knotted with a "weaver's knot" to insure a continuous running yarn for the next operation.

The final package previous to weaving is to cop or quill which is made on a special type of machine with compensating tension devices.

Glimpses Into the Past of the Dyeing Industry

(Continued from page 202)

The Pope was skeptical of Castro's story and sent men skilled in such matters to check his find, then still others to verify the favorable reports of the first. Final vindication of Castro's claims was brought about through an inspection by artisans from Genoa, formerly employed in the Turkish mines, who "having closely examined the nature of the place, declared it to be similar to that of the Asiatic mountains which produced alum; and shedding tears of joy, they knelt down three times, worshiping God, and praising His kindness in conferring so valuable gift on our age."

Operation of the mines commenced immediately and the alum was found to be far superior to that of Turkey. The Genoese purchased a quantity of it for which they paid twenty thousand pieces of gold and Cosmo de Medici was another early customer, paying to the Pope a total of seventy-five thousand pieces of gold. Castro was richly rewarded and the Pope erected a statue to him with the inscription: "To John de Castro, the inventor of alum."

Thus it fell to the lot of the ancient industry of dyeing to contribute largely to the support of the Christian religion during several centuries.

New du Pont Dyes

Pontamine Fast Brown 2RL, a new dyestuff claimed to be of exceptional fastness to light, has recently been developed by the Dyestuffs Division of E. I. du Pont de Nemours & Company. It is described as redder and brighter than Pontamine Fast Brown RKL, and can be used either as a self color or in combinations where a reddish brown component is indicated for the production of mode shades.

It is expected that the new color will find its chief use in the dyeing of cotton and rayon.

The Dyestuffs Division of E. I. du Pont de Nemours & Company has recently perfected Pontamine Diazo Violet RR, a developed color which, when diazotized and subsequently developed with beta naphthol, produces reddish shades of violet. It is described as redder and brighter than Pontamine Diazo Violet BL and, like the latter, is said to be particularly useful for dyeing cotton and rayon piece goods that are to be discharged.

Lowell Textile Alumni

A meeting of the New York Chapter of the Lowell Textile Institute Alumni was held on Tuesday evening March 14th, at the Happiness Restaurant on Fifth Avenue and 44th Street. Among other business transacted the annual election for officers for the ensuing year took place. Adolph J. Winkler, consulting textile chemist, was re-elected to the office of president. Other officers elected were as follows: vice-president, L. J. Dogin, American Aniline Products Co.; treasurer, C. C. Jessop, Louis Levy & Co.; and chairman entertainment committee, N. A. Johnson, Howes Publishing Co.

It was voted to hold another meeting the early part of May.

New Patents

(Abstracted by Synthetic Organic Chemical Manufacturers Association)

Manufacture of 1-Halogen-Aryl-Thiazoles. (Intermediate products for dyestuffs, for pharmaceutical preparations or for combating pests, as accelerators for vulcanization and the like.) Winfrid Hentrich and Max Hardtmann, of Wiesdorf-on-the-Rhine, Josef Hilger, of Cologne-Mulheim, and Werner Zerweck, of Frankfort-on-the-Main-Fechenheim, Ger., assignors to I. G. Farbenindustrie Aktiengesellschaft, of Frankfort,on-the-Main, Ger., No. 1,878,699, Sept. 20, 1932.

Monoazodyestuffs. (Dye wool even bluish-red to bluish-green shades of excellent fastness to fulling and light; for example, comprises coupling the sodium salt of 5-nitro-2-amino-1.1'-diphenyl-sulfone-3'-sulfonic acid with 2-methylamino-naphthalene-7-sulfonic acid.) Winfrid Hentrich, of Wiesdorf-on-the-Rhine, and Rudolf Knoche, of Leverkusen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,878,700, Sept. 20, 1932.

Water Soluble Leuco Esters of Vat Dyestuffs. (More specifically, comprises reacting upon leuco-1.2.2'.1'-dihydro-anthraquinone-azine with benzoic acid meta-sulfo chloride in the presence of pyridine.) Walter Mieg, of Opladen, near Cologne, and Rudolf M. Heidenreich, of Leverkusen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,878,964, Sept. 20, 1932.

Vat Dyestuff of the Anthraquinone Acridone Series. (Dyeing cotton even and fast brown to black shades.) Walter Mieg, of Opladen, and Robert Berliner, of Leverkusen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,878,965, Sept. 20 1932.

Wool-Dyestuff of the Dianthraquinonylamine Series. (Dyeing wool clear and strong greyish-blue to greenish-blue-grey shades of excellent fastness.) Walter Mieg, of Opladen, and Kurt Bamberger, of Elberfeld, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,878,966, Sept. 20, 1932.

Vat Dyestuffs of the Polynuclear Aromatic Imidazole Series and the Process of Preparing Them. (Dye cotton fast blue tints. Contains subject matter in common with Patent No. 1,715,430, dated June 4, 1929.) Maximilian Paul Schmidt, and Wilhelm Neugebauer, of Biebrich-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,878,986, Sept. 20, 1932.

New Water Soluble Sulfuric Acid Esters of Leuco Derivatives of Indigoid Vat Dyestuffs. (Suitable for dyeing and printing, yielding black, blue, violet, orange and corinth shades.) Wilhelm Bauer, Josef Haller, and Ludwig Zeh, of Wiesdorf-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,028, Sept. 27, 1932.

New Azodyestuffs. (Obtainable by diazotizing o-aminobenzoic acid and coupling with a diacetoacetyl derivative of 4.4'-diaminodiphenyl; dye vegetable fiber yellow shades fast to light and washing.) Max Hardtmann, of Naunhof, near Leipzig, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,223, Sept. 27, 1932.

Azo Dyestuffs. (Containing copper, dyeing vegetable fibers clear violet to blue shades fast to ironing, alkali and light.) Josef Hilger and Karl Wiedemann, of Cologne-Mulheim-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,240, Sept. 27, 1932.

Monoazo Dyestuffs. (Comprises diazotizing 4-nitro-1-aminobenzene-2-sulfoxy ethylanilide and coupling with 2-amino-8-hydroxynaphthalene-6-sulfonic acid; dye wool blue and Bordeaux red shades of good fastness properties.) Heinrich Hoyer and Eugen Glietenberg, of Leverkusen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,261, Sept. 27, 1932.

Watersoluble Diazo Compound. (For use in the manufacture of azo dyestuffs, comprises reacting upon methyl-imino-methyl-sulfurous acid with the diazo-compound from 4-chloro-2-toluidine, in aqueous solution, in the presence of the sodium salt of acetic acid.) Wilhelm Neelmeier, and Eugen Glietenberg, of Leverkusen, near Cologne, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,424, Sept. 27, 1932.

New Vat Dyestuffs of the Anthraquinone Series. (For example, comprises reacting upon 2-amino-3-cyanoanthraquinone in pyridine with cuprous cyanide; dye cotton yellow to violet shades of good fastness properties.) Robert E. Schmidt, Berthold Stein, and Kurt Bamberger, of Elberfeld, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,879,535, Sept. 27, 1932.

Azo Dyestuff and a Process of Preparation. (Comprises coupling a 1-(2'-5'-dichloro-p'-sulfo-)-phenyl-5-pyrazolone body with a tetrazetized 4-'-diamino-diphenyl-methane body; dye wool greenish yellowish tints to reddish orange tints fast to light and milling.) Daniel Zinner, of So. Milwaukee, Wis., assignor, by mesne assignments, to E. I. du Pont de Nemours & Co., No. 1,880,225, Oct. 4, 1932.

Azo Dyestuff and Process of Making the Same. (A continuation in part of above patent No. 1,880,225.) Daniel Zinner, of So. Milwaukee, Wis., assignor, by

mesne assignments, to E. I. du Pont de Nemours & Co., No. 1,880,226, Oct. 4, 1932.

Azo Dyestuff and Methods of Making Same. (More specifically, comprises coupling para-hydroxyl-diphenyl with a diazotized aromatic amino compound; yield on wool orange shades fast to to light and washing.) Ernest F. Grether, of Midland, Mich., assignor to the Dow Chemical Co., of Midland, Mich., No. 1,880,432, October 4, 1932.

Azo Dye and Method of Making Same. (A division of above patent No. 1,880,432.) Ernest F. Grether, of Midland, Mich., assignor to the Dow Chemical Co., of Midland, Mich., No. 1,880,443, October 4, 1932.

New Vat Dyestuffs of the Anthanthrone Series. (Obtained by reacting with dibromoanthanthrone upon 1-benzoylamino-4-aminoanthraquinone in the presence of naphthalene, anhydrous sodium acetate and copper acetate; dye cotton blue to grey to black shades.) Rudolph Heidenrich, of Leverkusen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,880,440, October 4, 1932.

Vat Dyestuffs of the Anthraquinone Series. (Obtainable by converting phenylene or naphthylene-di (1-thio-anthraquinone-2-carboxylic acids) into dithioxanthones; dye cotton orange to brownish red shades of good fastness properties.) Georg Rosch, of Cologne-Mulheim, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,880,490, October 4, 1932.

Preparation of Diazo Baths Suitable for the Production of Azo Dyestuffs on the Fiber. Carl Taube, of Leverkusen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,880,522, October 4, 1932.

Color Lake. (An improved process of producing color lakes which comprises precipitating a leuco compound of a basic dyestuff of the triarylmethane series with a solution of a complex inorganic acid.) Reinhard Buchwald, of Ludwigshafen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,880,761, October 4, 1932.

Process for Producing Nitroso Diazo Solutions. (To be used for dyeing purposes, and more particularly a process of preparing solutions containing N-nitroso-diazonium compounds of the diphenylamine series; yield valuable blue shades when combined on the fiber with 2.3-hydroxy-naphthoyl-arylamides.) Rudolph Jung, of Offenbach-on-the-Main, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,880,959, October 4, 1932.

Manufacture of Vat Dyestuffs of the Anthraquinone Series. (Obtained by condensing aldehydes of the anthraquinone series with amino-anthraquinones, the reaction products being azo-methines; dye cotton fast orange, yellow, olive, black, and claret red shades.) Max Albert Kunz, of Mannheim, Ger., assignor, by mesne assignments, to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,881,697, October 11, 1932.

Sulphur Containing Vat Dyestuffs and Process of Making Them. (Fast dyestuffs of great tinctorial power and various tints obtainable by transforming 2.5-diarylamino-3-mercapto-1.4-benzoquinones into the corresponding thiazine derivatives by subjecting them to an oxidation process.) Karl Thiess, of Frankfort-on-the-Main-Sindlingen, and Fritz Mannchen, of Frankfort-on-the-Main-Hochst, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,263, October 11, 1932.

Bromination Product of the Dibenzanthrone Series. (More specifically, mono-chloro-dibromo-dibenzanthrone, dyeing cotton fast blue shades.) Max Albert Kunz, of Mannheim, and Karl Koeberle, of Ludwigshafen-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,285, October 11, 1932.

Arylamides of Meta-Hydroxy-Diarylamine-Carboxylic Acids and Process of Making Same. (Valuable dyestuff intermediates.) Leopold Laska and Oskar Haller, of Offenbach-on-the-Main, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,286, October 11, 1932.

Manufacture of Water-Insoluble Azo Dyestuffs. (Either in substance, or on the fiber, yielding shades of red, blue and black.) Eugen Glietenberg, and Wilhelm Neelmeier, of Leverkusen-on-the-Rhine, and Josef Haller, of Wiesdorf-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,560, October 11, 1932.

Dyeing Preparation. (A water- or alkali-soluble composition for dyeing and printing, yielding yellow, orange, violet, red and blue shades.) Josef Haller, of Wiesdorf-on-the-Rhine, Eugen Glietenberg, of Leverkusen-on-the-Rhine, and Winfrid Hentrich, of Wiesdorf-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,561, October 11, 1932.

Water Soluble Diazo-Amino Compound. (Of value in the manufacture of dyestuffs and for combating insect pests.) Eugen Glietenberg and Wilhelm Neelmeier, of Leverkusen-on-the-Rhine, and Josef Haller, of Wiesdorf-on-the-Rhine, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,882,562, October 11, 1932.

New Mordants. (Alkali-metal salts of phenol-sulphur condensation products.) Moses L. Crossley and Maurice L. Dolt, of Plainfield, N. J., assignors to the Calco Chemical Co., Inc., of Bound Brook, N. J., No. 1,882,777, October 18, 1932.

New Mordants. (For use in fixing basic dyes in paper.) Moses L. Crossley and Maurice L. Dolt, of Plainfield, N. J., assignors to the Calco Chemical Co., Inc., of Bound Brook, N. J., No. 1,882,778, October 18, 1932.

Manufacture of Azo Dyestuffs. (Prepared by diazotizing an anthraquinone derivative containing a diazotizable amino group and coupling with an amino naphthoic acid or a side-chain or nuclear substitution product thereof; yield red to brown shades.) George Holland Ellis, of Spondon, near Derby, Eng., assignor to Celanese Corp. of America, No. 1,883,356, October 18, 1932.

Chloro-Anthraquinone-Acridones and Process of Making Same. (Valuable red vat dyestuffs.) Walter Bruck, of Mannheim, Ger., assignor to I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-the-Main, Ger., No. 1,883,525, October 18, 1932.

Pyranthrone Vat Dye and Method of Making Same. (Suitable for dyeing textile fibers, especially cotton, brown shades.) Wilfred M. Murch, of Hamburg, N. Y., assignor to National Aniline & Chemical Co., Inc., of N. Y., N. Y., No. 1,884,122, October 25, 1932.

Process of Producing Sulphur Dyes. (Comprises passing the vapors of a volatile organic compound containing hydrogen through molten sulphur; yield brown to green shades on cotton). Stewart J. Lloyd and George D. Palmer, Jr., of University, Ala., assignors of one-third to Absalom M. Kennedy, of University, Ala., No. 1,884,762, October 25, 1932.

Easily Soluble Dyestuffs of the Anthraquinone Series. (Comprises reacting beta-halogen derivatives of arylaminoanthraquinones heteronuclearly sulphonated in alpha- and beta-position with sulphite in presence of water; dye wool and silk blue tints of excellent fastness, suitable also for printing purposes.) Samuel von Allmen, of Basel, Switzerland, assignor to the Firm: Chemical Works, Formerly Sandoz, of Basel, Switzerland, No. 1,885,065, October 25, 1932.

Vat Dyestuff and Process of Making Same. (Comprises condensing a 1.4.5.8-naphthalenetetra-carboxylic acid with an ortho-diamine; dyes cotton green and when the material is exposed to the air, changes to a grey to black and also reddish brown tint.) Wilhelm Eckert and Heinrich Greune, of Frankfort-on-the-Main-Hochst, Ger., assignor to General Aniline Wks., Inc., Original Pat. No. 1,588,451, dated June 15, 1926, Serial No, 44,-369, filed July 17, 1925, Re. 18,643, Nov. 1, 1932.

New Derivatives of 1.2.7.8-Diphthaloylcarbazole. (Dye cotton orange to red to red-brown shades of excellent clearness and very good fastness to light and washing. A continuation-in-part of U. S. Patent No. 1,730,186, dated Oct. 1, 1929.) Fritz Baumann, of Leverkusen-on-the-Rhine, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,885,172, Nov. 1, 1932.

Coloring Matters and Intermediates of the Benzanthrone Series. (Consists of treating the condensation product from benzanthrone and formaldehyde in sulphuric acid, with benzoyl chloride, aluminum chloride, sodium chloride and an oxidizing agent, yielding a new bright yellow product soluble in alkali.) David Clarence Rhys Jones, of Polmont, Scotland, Robert Fraser Thomson, of Carlisle, England, and John Thomas, of Polmont, Scotland, assignors to Imperial Chemical Industries, Ltd., No. 1,885,449, Nov. 1, 1932.

Process for the Manufacture of New Xanthene Dyes. (Comprises condensing a 3:7-tetra-alkyldiamino-xanthone with para-tolyl-meta-phenetidine, in the presence of phosphoryl chloride; dye animal fibers, mordanted cotton and cellulose ester and other materials, in bright bluish violet shades which are characterized by their remarkable blue fluorescence in ultra violet light.) Albert Coulthard and Ernest Harry Rodd, of Blackley, Manchester, Eng., assignors to Imperial Chemical Industries, Ltd., of London, Eng., No. 1,885,591, Nov. 1, 1932.

Process for the Manufacture of New Xanthene Dyes. (A division of above patent No. 1,885,591). Albert Courthard and Ernest Harry Rodd, of Blackley, Manchester, England, assignors to Imperial Chemical Industries, Ltd., of London, Eng., No. 1,885,592, Nov. 1, 1932.

Manufacture of Polyazo Dyestuffs. (Consists in combining in mineral acid solution tetrazotized benzodine with 1:8-aminonaphthol-3:6-disulphonic acid and then coupling with 1-diazonaphthalene-4-sulphonic acid and N-(o-tolyl-)m-aminophenol; dye artificial silk, wool, natural silk and leather in level black shades.) Hans Leemann, Heinrich Lier, and Oskar Knecht, of Basel, Switzerland, assignors to Chemical Works formerly Sandoz, of Basel, Switzerland, No. 1,885,609, November 1, 1932.

Dyestuffs Containing Metals and Process of Making Same. (Derived from the nitrated diazo-compound of 1-amino-2-hydroxynaphthalene-4-sulfonic acid; dye fibers of all kinds fast black shades and also suitable for coloring varnishes.) Fritz Straub, of Basel, and Hermann Schneider, of Riehen, near Basle, Switzerland, assignors to Society of Chemical Industry in Basle, of Basel, Switzerland, No. 1,885,641, Nov. 1, 1932.

New Sulphur-Dyestuffs and a Process of Preparing Them. (Obtainable by melting apphenazine compound with an alkali metal polysulphide in the presence of a compound of a metal of the group consisting of molybdenum, tungsten, vanadium, uranium and antimony; yield on cotton bluish green, brown and red tints.) Otto Bayer, of Frankfort-on-the-Main, Wilhelm Hohenhemser, of Frankfort-on-the-Main-Fechenheim, and Karl Larbig, of Frankfort-on-the-Main-Hochst, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,886,365, Nov. 8, 1932.

Dyestuff Containing Cellulose Residues and Process of Making Same. (As new products the cellulose derivatives which contain simultaneously a cellulose residue, a cyanuric ring and a residue of an azo-dyestuff of the benzene and naphthalene series.) Robert Haller, of Riehen, near Basel, and Alphons Heckendorn, of Basel, Switzerland, assignors to Society of Chemical Industry in Basle, of Basel, Switzerland, No. 1,886,480, Nov. 8, 1932.

Textile Foundation News

(Continued from page 228)

"To produce a specific dye, a certain arrangement of particular substituents is necessary. Even when the desired arrangement is obtainable, others are likely to occur simultaneously, causing a considerable waste in the manufacturing process. Obviously, then, it is of great interest

to the producer to control and direct effectively the addition to the benzene ring of the desired substituents.

"Among the substituents commonly introduced there is one whose entry is effected by sulfonation, a process important in manufacturing many dyes and also in the study of orienting influences. Hence, our research is investigating the sulfonation of benzoic acid, a substance closely related to benzene and differing from it only in the attachment of a well-known substituent to one of the six points.

"In the process of sulfonation a second and different substituent is added to one of the six points. In order to explain how and where the second substituent is added, it is desirable to know how fast the process occurs, another object of our research. We hope from these studies to contribute to an effective control of the point of entry of substituents to the benzene ring, thereby leading to more economical production of well-known dyes and possibly to the development of new ones."

Rise in the Clouding-point in Soap Solutions

Deutscher Faerber Ztg. 68, 74 (1933) (*Communication No. 291 in the Technical Notes.*)—In reference to the temperature at which turbidity sets in, the "clouding-point," of a whole series of good Marseilles soaps of different origins and of different ages, certain irregularities have been detected whose explanation was not at first possible. Soaps which were manufactured from the same materials at the same time of the year displayed striking deviations in their clouding-point, one from another; the soaps, e.g., manufactured in summer, showing a higher clouding-point than those produced during the winter months. The age, too, of the soap appeared to have some influence, for a freshly-prepared soap, with a clouding-point of 17°, after 10 days displayed a clouding-point of 32°. In the determinations carried out, the details of the method were, of course, maintained uniform, it being well known that rapid cooling gives a lower clouding-point, and the lower, the more rapid the cooling. Accordingly, cooling the soap solutions in a water or ice bath gives false results; the method consisted in allowing the solutions to cool spontaneously in the laboratory, until the point of turbidity was reached. It is a striking fact, and one apparently not yet noted in the literature, that the clouding-point rises decidedly not only by slow cooling, but also by mere long standing of the solution under examination. This rise in the clouding-point becomes especially important, when one repeatedly warms and cools the same solution. Through this simple treatment, the clouding-point of some solutions rose in a short time from 26° to 55°. This last fact seems to be of great significance in respect to the industrial use of standing soap baths or of stock solutions. In those industries employing such baths, as in silk dyeing and bleaching, it should not be forgotten that such solutions, even in the shortest time, will display a clouding-point quite different from, and higher than, the original value. According to our investigations, the possibility is not excluded, that, with specially sensitive goods, such as weighted silks, the same difficulties will arise from the use of soap baths with an originally low clouding-point, but repeatedly used, as will come from the immediate use of a bath from soap which at the first possessed a high clouding-point.

Bradford-Durfee Alumni

New York and New Jersey Alumni of the Bradford-Durfee Textile School, Fall River, Mass., met Friday, February 24th at the Hotel Bristol, West Forty-Eighth Street, New York, to discuss the forming of a metropolitan alumni association of the school.

Twenty-four graduates of the school attended the meeting and it was unanimously decided to form a metropolitan alumni association and to hold regular get-togethers every six to eight weeks.

The following officers were elected:

President—Arthur E. Jerome, class of 1922, Massasoit Mfg. Co.

Secretary—James L. Giblin, class of 1925, United States Testing Co., Inc.

Treasurer—Charles L. Lubinsky, class of 1925, Bear Mills Mfg. Co.

The next meeting of the Alumni association will be held on Friday, April 7, 6:30 p. m. at the Cabin Grill, 490 Seventh Avenue, between 36th and 37th Streets. It is hoped that every graduate in the New York and New Jersey districts will be present.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

GRADUATE: Textile Student, age 24; three years large finishing plant, color shop experience. Experienced with fast colors on cotton and rayon; also with silk colors. References. Address Box No. 772, American Dyestuff Reporter, 440 Fourth Avenue, New York.

CHEMIST with 7 years' laboratory and practical mill experience in scouring, spinning and dyeing, and with a thorough knowledge of all types of dyestuffs, is desirous of connecting with a progressive woolen or carpet mill as chemist. Salary no object until worth is demonstrated. Box No. 773, American Dyestuff Reporter, 440 Fourth Avenue, New York.